

TO STUDY THE CHANGES IN THE MECHANICAL PROPERTIES OF OFHC COPPER DURING BRAZING

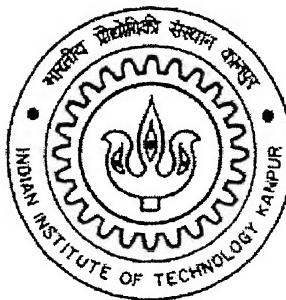
A Thesis Submitted
In Partial Fulfillment of the Requirements
for the degree of

MASTER OF TECHNOLOGY
in
MATERIALS AND METALLURGICAL ENGINEERING

by

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INDIAN INSTITUTE OF TECHNOLOGY, KANPUR
JULY 2004

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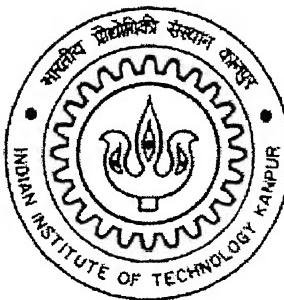
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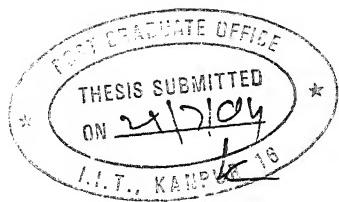
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JULY 2004



CERTIFICATE

This is to certify that the work contained in the thesis entitled "**To study the changes in the mechanical properties of OFHC copper during brazing**" by Mr. Aniruddha Bose has been carried out under our guidance and that this work has not been submitted elsewhere for a degree.

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ABSTRACT

A Radio Frequency Quadrupole (RFQ) structure is being proposed to be built in C.A.T Indore by brazing together pieces (1metre long) of OFHC copper, using hydrogen furnace operated at temperatures above $0.75T_m$ (T_m is the melting point of OFHC copper in °K). The requirements of this structure are that the brazed material must be strong mechanically after the brazing operation and that the joints should be leak-tight. The deterioration of the mechanical properties of OFHC copper due to heat of brazing was studied by measuring the changes in yield strength, ultimate tensile strength and hardness values at different temperatures by simulating the brazing conditions that would be followed later on. Two different heating rates (150°C/hr and 700°C/hr) were used in the simulation. Brazing of OFHC copper (at high temperatures) necessarily caused annealing and grain growth, resulting in a decrement of mechanical properties. It was observed that the decrease in tensile strength was greater in case of higher heating rates. However the decrease in strength in both cases was appreciable only above 900°C, hence in the subsequent brazing procedure copper-silver eutectic having a melting point of 780°C was chosen as the filler metal. In addition to tensile testing and shear strength measurement were also done on brazed samples. It was observed that the failure invariably occurred in the base metal indicating much higher joint strength. Finally, the flow of filler metal through the joint was investigated and it was found that the flow depended on several criteria, the most important being the cleanliness of the faying surfaces. Leak tightness of 10^{-10} atm/cm³/sec of the brazed joints was also achieved.

CONTENTS

LIST OF FIGURES.....	i
LIST OF TABLES.....	v
1. INTRODUCTION.....	1
2. LITERATURE SURVEY.....	3
2.1 RADIO FREQUENCY QUADRUPOLE.....	3
2.1.1. PRINCIPLE OF OPERATION.....	4
2.1.2. FABRICATION OF RFQ.....	5
2.1.2.1 MODES OF FABRICATION ALREADY TESTED.....	6
2.2 OBJECTIVES OF WORK.....	11
2.3 OFHC COPPER.....	12
2.3.1. COMPATIBILITY OF OFHC COPPER FOR THE RFQ STRUCTURE.....	13
2.3.2. EARLIER WORK DONE ON OFHC COPPER.....	14
2.4 BRAZING AND ITS PRINCIPLES.....	17
2.4.1. BACKGROUND.....	17
2.4.2. PARAMETERS AFFECTING BRAZING.....	18
2.4.2.1. SURFACE ENERGY AND SURFACE TENSION.....	19
2.4.2.2. WETTING AND CONTACT ANGLE	20
2.4.2.3. FLUID FLOW.....	23
2.4.2.4. FILLER SPREADING CHARACTERISTICS.....	24
2.4.2.5. SURFACE ROUGHNESS OF COMPONENTS.....	26

2.4.2.6. DISSOLUTION OF PARENT MATERIALS BY FILLER METAL.....	27
2.4.2.7. SIGNIFICANCE OF THE JOINT GAP.....	29
2.4.3. FUNCTIONAL AND DESIGN REQUIREMENTS.....	33
2.4.3.1. METALLURGICAL STABILITY.....	33
2.4.3.2. MECHANICAL INTEGRITY.....	33
2.4.3.3. ENVIRONMENTAL DURABILITY.....	33
2.4.3.4. ELECTRICAL AND THERMAL CONDUCTIVITY.....	34
2.4.4. PROCESSING ASPECTS.....	34
2.4.4.1. JIGGING OF THE COMPONENTS.....	34
2.4.4.2. FORM OF THE FILLER METAL.....	35
2.4.4.3. HEATING METHODS.....	35
2.4.4.4. TEMPERATURE MEASUREMENT.....	35
2.4.4.5. JOINING ATMOSPHERE.....	36
2.4.4.6. CLEANING TREATMENTS.....	37
2.4.4.7. HEATING CYCLE OF THE JOINING OPERATION.....	37
3. EXPERIMENTAL PROCEDURE.....	38
3.1 STUDY OF DEGRADATION OF MECHANICAL PROPERTIES OF OFHC COPPER AFTER BRAZING	38
3.1.1. STEPS IN EXPERIMENTAL WORK.....	39
3.1.2. EVALUATION PROCESSES OF THE SAMPLES.....	44
3.2 STUDY OF THE BRAZING PROCESS OF OFHC COPPER.....	45
3.2.1. STEPS IN EXPERIMENTAL WORK.....	45
3.2.2. TESTING OF THE SAMPLES.....	48

3.2.3. EVALUATION PROCESSES OF THE BRAZED SAMPLES.....	54
4. RESULTS AND DISCUSSION	
4.1 MECHANICAL PROPERTY DETERIORATION.....	55
4.2 RESULTS OBTAINED ON BRAZED SAMPLES.....	62
5. CONCLUSIONS.....	82
REFERENCES.....	84

LIST OF FIGURES

Figure No.	Figure Caption	Page
2.1.	Layout of the linac	3
2.2.	Schematic of the Radio Frequency Quadrupole	4
2.3.	Assembly of RFQ structure	5
2.4.	Machined Segment of RFQ module using brazing	5
2.5.	A brazed 0.45 MeV RFQ	7
2.6.	Vane subassembly section	8
2.7.	Quadrant sub-assembly scheme	8
2.8.	Major Vane sub-assembly scheme	9
2.9.	Pre braze tuning	9
2.10.	Segment final braze assembly	10
2.11.	A brazed RFQ structure at KEK, Japan	14
2.12.	Surface after HPR at $65\text{kg}/\text{cm}^2$ for 2 hours	15
2.13.	Copper surface after brazing heat cycle in hydrogen furnace	15
2.14.	Copper surface after diffusion bonding heat cycle in vacuum furnace	16
2.15.	Schematic representation of braze joint	17
2.16.	Simplified diagrams of surface energies. Atom B at the surface has saturated bonds and thus a higher energy than atom A. This difference in energy is the origin of γ_{sv}	19
2.17.	Schematic diagram used to explain the relationships between surface energy and surface tension	20
2.18.	Surface tension forces acting when a liquid droplet wets a solid surface, according to the classical model	21
2.19.	Rise of a liquid between two parallel plates	22
2.20.	Results of a sessile test to analyze spreadablilty as a function of the solid and liquid composition	25

2.21.	The concentration of solid metal in a liquid metal wetted by it changes in an inverse exponential manner with respect to time and is limited by the saturation concentration of the solid constituent in the liquid at that temperature	29
3.1	It shows the furnaces used for the experiments. The top one is the larger Furnace (CAT Indore) while the bottom one is the smaller furnace (IIT Kanpur)	41
3.2	The heating cycle of larger cylindrical furnace at CAT Indore	43
3.3	The heating cycle of small cylindrical furnace at IIT Kanpur	43
3.4	The heating schedule followed during the actual brazing process	48
3.5	The schematic of the cubic sample	49
3.6	Design of tensile samples	
3.6.1	Design of tensile sample TT1B 850	50
3.6.2	Design of tensile sample TT1S 850	50
3.7	The design of tensile samples TT2S 830 and TT3S 830 with wire as filler metal. Note that design tolerances were applied	51
3.8	Small prototype of the tensile sample which was brazed to study the flow of filler metal by Scanning electron microscopy	52
3.9	The sample that was used for testing the leak rate tightness of the joints in the tensile sample	53
3.10	The shear testing sample. Here OD means overlap distance	54
4.1	The tensile strength variation with temperature is shown for the first set of experiments done at CAT Indore	58
4.2	The tensile strength variation with temperature is shown for the first set of experiments done at IIT Kanpur	59
4.3	The range of Vicker's hardness values at different temperatures	60
4.4	The figure shows grain growth with temperature at a magnification of 500X	61
4.5	Butt joint failure during tension test as seen in SEM. No flow of filler metal inside the joint.	62

4.6	The brazed TT1B 850C is shown in figure where the total filler metal has overflowed from the joint	64
4.7	The optical micrograph shows good flow of filler metal at a magnification of 200X. The photograph was taken after etching it with aqueous ferric chloride	65
4.8	The optical micrograph shows no flow of filler metal at all through the joint. The photograph was taken without etching	66
4.9	The optical micrograph shows no flow of filler metal at all through the joint. The photograph was taken after etching	66
4.10	The Scanning Electron micrograph shows good flow of filler metal through the joint. The photograph was taken after etching	67
4.11	The Scanning Electron micrograph shows no flow of filler metal (even at 1000X) at all through the joint. The photograph was taken after etching	67
4.12	The optical micrograph shows good flow of filler metal at a magnification of 500X. The photograph was taken after etching it with ferric chloride	69
4.13	The optical micrograph shows good flow of filler metal at a magnification of 500X. The photograph was taken without etching	69
4.14	The SEM photo shows uniform flow of filler metal through the joint. The sample was slightly etched	70
4.15	The SEM photo shows almost uniform flow of filler metal at another location of the sample. The sample was slightly etched	71
4.16	SEM photo of the fractured surface of TT1S 840	73
4.17	SEM photo of the fractured surface of TT2S 830 which failed through ductile mode fracture from the base metal	73
4.18	SEM photo of the fractured surface of TT2S 830 which failed through ductile mode fracture from the base metal	74
4.19	SEM photo of the interface of the base metal and the filler metal of a small prototype of the tensile sample	75

4.20	The compositional analysis of point 1 gives the actual composition of the filler metal	76
4.21	The compositional analysis of point 2 which suggests that there has been some diffusion of copper into the filler metal.	77
4.22	The hardness profile across the interface is shown. The shaded portion shows the interface region	78
4.23	A and B are the positions where the samples were held in the clamps of the tensile testing machine	79
4.24	Graph showing the minimum load upto which the joint can withstand shear load. In each case the failure occurred at the base metal. Although some tearing was observed at the joint when at minimum overlap distance	80
4.25	The shear sample having overlap distance of 2T has failed from the base metal although some tearing at the joints are seen	81

LIST OF TABLES

Table No.	Table Caption	Page
2.1	Properties of OFHC copper	12
2.2	Comparison of soldering, brazing, and welding [Metals Handbook Vol 6 1983]	18
2.3	Recommended joint clearance at brazing temperature [Brazing Handbook, 1991]	31
4.1	Tensile strength evaluation done in CAT Indore	55
4.2	Tensile strength evaluation done in IIT Kanpur	56
4.3	Hardness variation with temperature IIT Kanpur	57
4.4	Results of tensile testing of brazed samples	72
4.5	Compositional analysis at points 1 and 2.	78

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CHAPTER 1

INTRODUCTION

A Radio Frequency Quadrupole structure (RFQ) is a low energy and high current accelerator for H⁺ and heavy ions. This model of accelerators achieves simultaneous focusing, bunching, and acceleration of the beam. As a result this type of structure helped in replacing the large DC accelerators as the pre-accelerator which required handling of very high voltages.

This RFQ structure (of its first kind) is being proposed to be built in the Centre of Advanced Technology (CAT), Indore, India. The structure is of the shape of a cylinder with octagonal cross section. The length of the structure is about 5 meters with a cross sectional width of 0.22 meter. The primary requirements of the material to be used for the RFQ are its high thermal and electrical conductivity. The best material satisfying these conditions is oxygen free high conductivity copper (OFHC). The only problem associated with this material is its low mechanical strength especially at high temperature. The fabrication of this heavy quadrupole structure, made of OFHC copper, requires joining of several pieces, each 1 meter length, preferably by brazing. Brazing offers a distinct advantage over other modes of fabrication since OFHC copper can be easily brazed and it also provides the desired leak tightness of joints which is the most important criterion of a RFQ structure. Each of these 1 meter pieces is divided into four quadrupole sections which also have to be brazed. During brazing there would be a definite deterioration in the mechanical properties of the material, because of the heat of brazing. The present study incorporates:

- i) An investigation into the extent of deterioration of mechanical properties of OFHC copper during brazing and its remedial measures.
- ii) Characterization of the OFHC copper samples brazed with copper-silver eutectic as the filler metal.

The ensuing chapters in this dissertation would try to explain the work that was done in a step by step manner. The chapter after the introduction part is “Literature review”, which provides a thorough background about the RFQ structure, the principles of brazing, OFHC copper and the objective of the work. The next chapter “Experimental procedures” explains

he processes involved in the study of deterioration of mechanical properties of OFHC copper and the characterization of the brazed samples. The next chapter deals with the results obtained and their discussion. The chapter following is the Conclusions part which summarizes the results of the experiments in a brief manner. The last chapter provides the relevant references for this thesis work.

CHAPTER 2

LITERATURE SURVEY

2.1. RADIO FREQUENCY QUADRUPOLE

The science and technology of proton accelerators have progressed considerably in the past three decades. Three to four orders of magnitude increase in both peak intensity and average flux has made it possible to construct high intensity proton accelerators for modern applications such as: spallation neutron sources, kaon factory, accelerator production of tritium, energy amplifier and muon collider drivers [W.T.Weng, 1997].

Apart from other sources contributing to the progress of proton accelerators the development of Radio frequency quadrupole (RFQ), replacement of Cockcroft-Waton as the pre-accelerator, is seen as one of the dominant factors. The high intensity proton accelerator has tremendous applications which would be discussed briefly at the end of this chapter. To elaborate on the importance of RFQ, a layout of a spallation neutron source is shown in Fig 2.1. The position of RFQ is shown in a box [S.A.Pande, 2004].

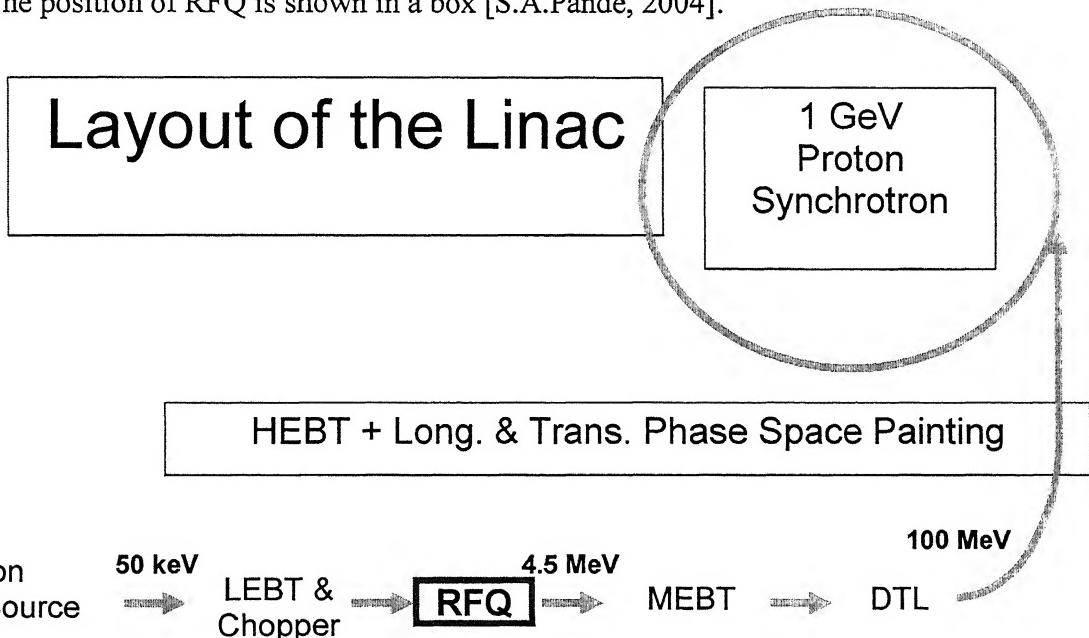


Figure 2.1: Layout of the linac [S.A.Pande, 2004]

DTL: Drift tube linac; **LEBT:** Low energy beam transport; **HEBT:** High energy beam transport

2.1.1 Principle of operation

A Radio Frequency Quadrupole is a four pole structure which is a low energy and high current accelerator for H^+ and heavy ions. This model of accelerators achieved simultaneous focusing, bunching and acceleration of the beam. This in turn replaced the earlier DC accelerators which required handling of very high voltages.

The focusing of the beam results due to the quadrupole structure while bunching is inherent as the two pairs of electrode are 90° apart in phase and the acceleration is due to the undulation of the pole tip. A schematic diagram of the RFQ along with its operating principle will be explained as follows.

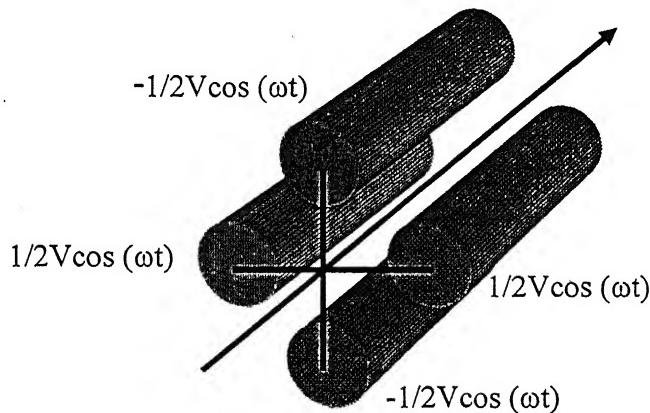


Figure 2.2: Schematic of the Radio Frequency Quadrupole

As the name suggests RFQ provides electric quadrupole focusing with electric field oscillating at radio frequency. The figure shows four equispaced electrodes with alternating polarity as we move from one to the other, thereby forming an electric quadrupole. The off axis particles will experience a transverse force which is alternating in time and this transverse force provides ‘Alternating Gradient’ focusing. The advantage of RFQ is that it provides electric focusing for low velocity particles which is stronger than conventional magnetic focusing. A structure with uniform electrodes along its length will have no component of electric field along the axis and thus will not work as an accelerator. To generate an axial electric field component, the quadrupole electrodes are modulated

longitudinally. One pair of electrodes is shifted longitudinally with respect to the other pair by 180° so that when the distance from the axis of vertical vanes is at its minimum ‘a’, the horizontal vanes will be maximum apart at ‘ma’. The axial electric field component is generated due to the potential difference between the point of minimum separation from axis of vertical vanes (or horizontal vanes) and the point of minimum separation from the axis of the horizontal vane (or vertical vane). In RFQ, the field in successive gaps is in opposite direction and therefore when it is accelerating in one cell, it is decelerating in the next. There are two unit cells per structure period. At a given time every alternate cell will have a particle bunch. In brief this is the working principle of the RFQ structure.

2.1.2 Fabrication of RFQ

This RFQ structure of its first kind is being proposed in Centre of Advanced Technology, Indore. This structure has an octagonal cylinder like shape having a length of 5 meters and a diagonal length of 0.22 meter approximately. The fabrication of this heavy structure in OFHC copper is to be done by brazing pieces of 1meter length. Moreover each quadrupole is made up of four sections which are to be joined by brazing. The figure below shows 1m section and one part of that quadrupole.

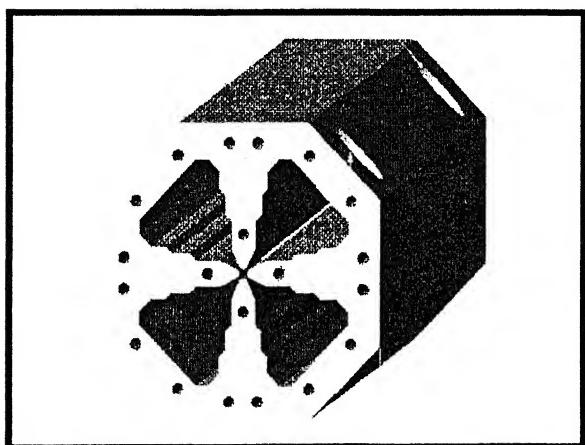


Figure 2.3: Assembly of RFQ structure module using brazing

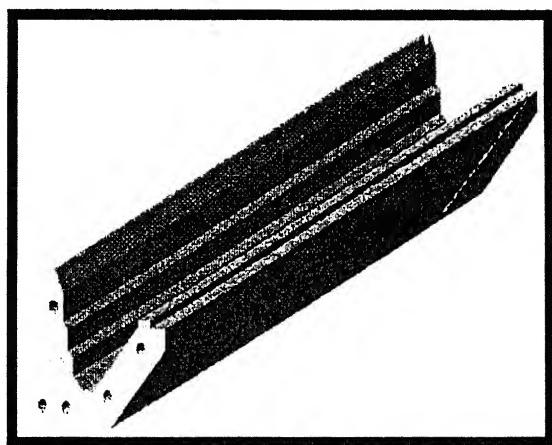


Figure 2.4: Machined Segment of RFQ

OFHC copper is oxygen free high conductivity copper whose density is 8.97gm/cm^3 . Thus the RFQ structure would be very heavy and difficult to braze. All over the world this structure is mostly made up of OFHC copper because of its very high thermal and electrical conductivity. The primary requirements for a RFQ material are its very high electrical and thermal conductivity. The functional requisites of an RFQ structure is its structural integrity and highly polished mirror like inner surfaces. Slight variations in the actual structure from the design would make the RFQ ineffective. The maximum tolerance that can be sustained along the length as reported is 0.1mm/m [M.Painchault et al. 2000]. Thus the requirements being that stringent means brazing should be almost perfect. Hence to achieve that perfection, a detailed study of this material is undertaken. After that some prototypes are brazed and joint quality is tested.

One of the main requirements of RFQ structure apart from thermal and electrical conductivity is its vacuum compatibility. It is impossible to fabricate this structure from a single piece, so it becomes evident that small pieces need to be joined. As a result, there would be number of joints in a single structure. Thus leak tightness of each of the joint assumes a critical significance. This leak tightness can only be offered by brazing process. That is the reason why no other processes for fabricating a RFQ structure is followed.

2.1.2.1 Modes of fabrication already tested

There are number of research labs where the study to develop and fabricate the RFQ structure is being carried out. Since the research is of high importance for any country the fabrication process is a closely guarded secret, although there are some approaches that have been mentioned. Those details of fabrication processes as followed by other laboratories are mentioned as follows.

In the project undertaken by LEDA in France, the RFQ to be built is of 8m length. Their primary study, before the fabrication process, involved various test for machining and brazing of copper. Their chief importance was linked to the grain size of the copper material. It is mentioned that for brazing operation a homogeneous copper in three directions is required. Since properties of copper are strongly dependent on its history, so the initial processing of copper assumes importance. So to characterize copper, hardness, a property easier to measure was utilized [M.Painchault et al. 2000].

The project named KOMAC (Korea Multi-purpose Accelerator Complex) [Choi et al. 1999] was undertaken by KAERI (Korea Atomic Energy Research Institute). The RFQ is 324 cm long, 4 vanes type. The RFQ is machined of OFHC copper; integrated from separate four sections which are constructed by using vacuum brazing. In fact it has been stated that the brazing process is the most difficult part of the fabrication process. It has also been noted that because of the leak of the brazing surface and the strain of the RFQ structure by furnace heat, it is important to determine the appropriate shape of the brazing area. To determine this, a prototype brazing on 96.4cm long, 0.45 MeV RFQ has been performed. The RFQ was brazed in a vacuum furnace with LUCAS BVAg-8, Ag-Cu alloy with a liquid temperature of 780°C. Testing of the brazed joint showed it to be leak tight. The brazed test piece is shown below.

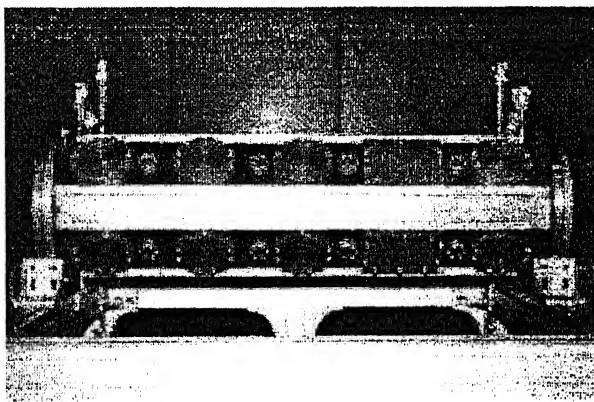


Figure 2.5: A brazed 0.45 MeV RFQ

There are other modes of fabrication which are still being studied. One such process is electroforming which is under consideration at ITEP Moscow. The present status of their fabrication approach can be broken down into three primary schemes:

- i) In the first scheme, machined and brazed vane details would be mechanically joined to a cavity cylinder with a welded and/or electroformed RF joint.
- ii) The second scheme would employ electroformed assembly of machined and brazed vanes into a cavity cylinder or possibly a fully electroformed cavity cylinder.

- iii) The third scheme that was ultimately chosen for the preliminary design effort is to have a fully brazed assembly using step brazing to fabricate the vanes and quadrant details and finally complete segment with the end flanges.

The RFQ is designed as a 100% OFE copper with either Glidcop or 304 SST end flanges. This approach was heavily borrowed from the techniques used successfully on the LEDA RFQ at Los Alamos [Scharge et al. 1998]. The fabrication process begins with the machining of the cooling channels into over sized OFE copper blanks and performing high temperature braze for assembly. Fig 2.6 and 2.7 illustrate the process for the vane blanks and for the quadrant blanks. They planned to use 35-65 Au-Cu alloy at 1027°C for the first braze step.

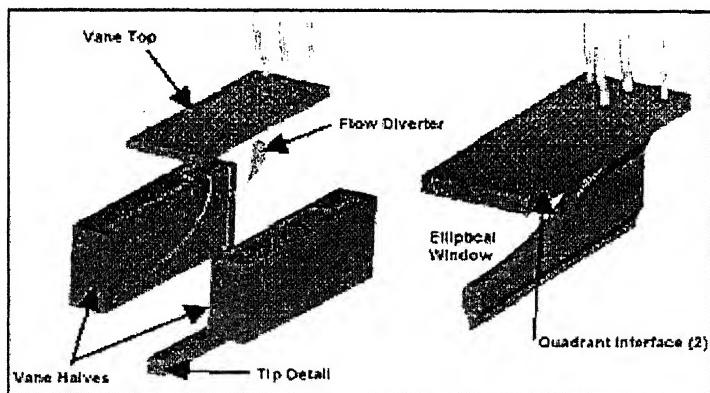


Figure 2.6: Vane subassembly section

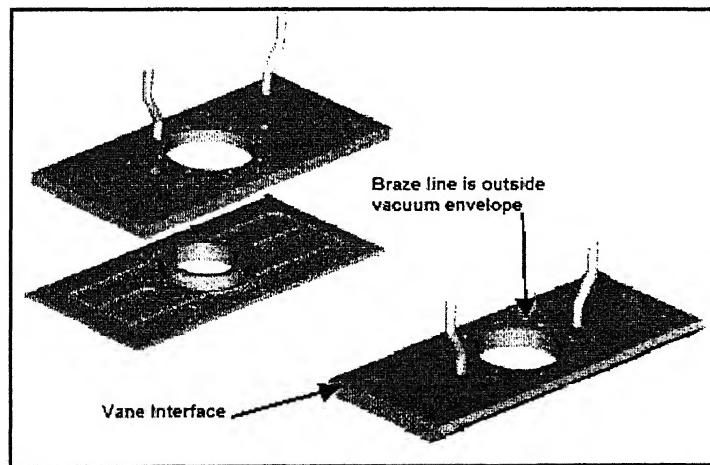


Figure 2.7: Quadrant subassembly scheme

With the cooling channel braze step complete, the detailed parts are machined in preparation for the next assembly step. The minor vanes are machined to their finished condition and are ready for segment assembly and tuning. This includes finishing of all the surfaces including the modulated vane tip. The only surfaces left oversized are the braze surfaces for connection to the cavity walls. These surfaces are machined during the tuning step. Prior to the final braze step the elliptical cut out in the vane is finished along with the braze contact surfaces on both the vane detail and the quadrant details. After brazing with 50-50 Au-Cu at 996°C the remaining surfaces are machined.

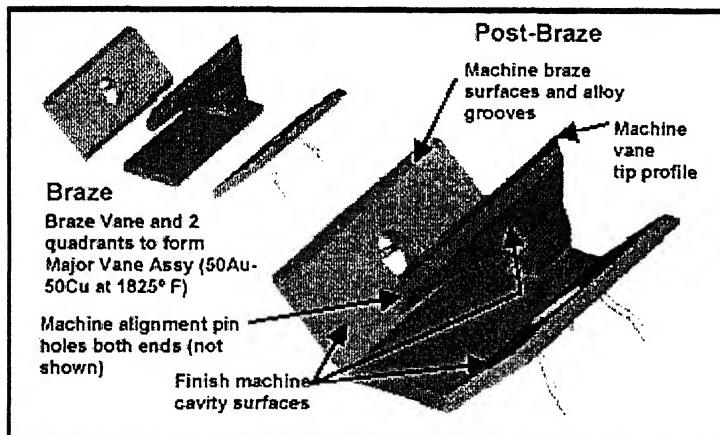


Figure 2.8: Major Vane subassembly scheme

At this point the two major and minor vanes are mounted on the fixture after machining for the final braze step. Fig 2.9 and 2.10 illustrates the final brazing step with 72-28 Ag-Cu 790°C. This alloy has the desirable property of thoroughly wetting a zero clearance joint.

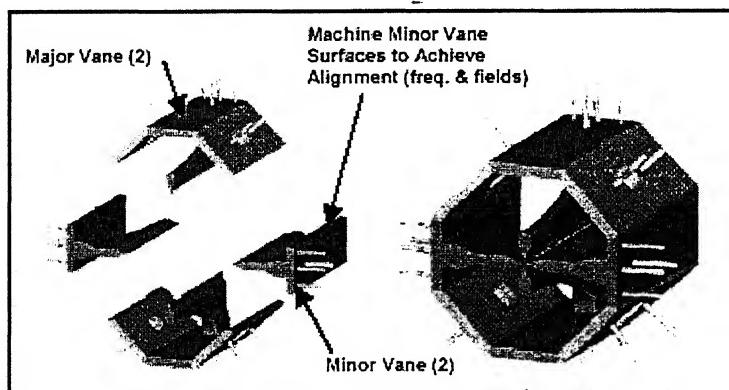


Figure 2.9: Pre braze tuning

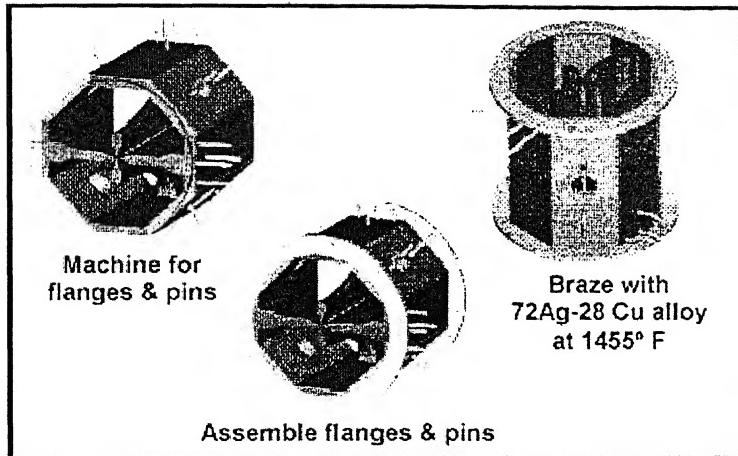


Figure 2.10: Segment final braze assembly

These works already done would later on serve the purpose of helping us in fabricating the RFQ structure. At present we would be concentrating on the evaluation of the deterioration of the mechanical properties of OFHC copper during high temperature brazing and one of the brazing of a few small copper samples. The above ideas in a nutshell can be summarized below and will be followed in our fabrication process.

- i) To study the changes in properties of OFHC copper due to high temperature brazing.
- ii) To check the grain homogeneity and the hardness values to understand the history behind the fabrication of the material.
- iii) To carry out brazing on small pieces of copper using preferably BAg-8 filler metal that melts at 780°C.
- iv) The furnace used mostly in brazing OFHC copper is vacuum furnace. However in our case we have preferred hydrogen furnace since chances of overflow of filler is greater in vacuum furnace.
- v) To check the leak tightness of each joint after brazing.
- vi) Even step brazing is a possibility [J.W.Rathke et al. 2002].

2. OBJECTIVES OF WORK

The ultimate objective of this work in the next few years would be the fabrication of a brazed RFQ structure preferably out of OFHC copper. However, as of now, the work is limited to the following investigations.

- i) To study the changes in mechanical properties of OFHC copper due to brazing at high temperature. The properties include the changes in yield and tensile strength, the increase in the range of variation in the hardness values, and the changes in grain size. It is but obvious that during brazing, the temperature would be well above the recrystallization and grain growth temperature of OFHC copper. This would definitely lead to the deterioration of the mechanical properties. So an attempt will be made to investigate the amount of deterioration and identify ways to prevent it.
- ii) The second task is to develop a procedure to braze small samples of OFHC copper using preferably copper-silver eutectic filler alloy. Moreover, the characterization of the brazed joint also needs to be done. This includes evaluation of the tensile and shear strength of the brazed joints; study of the flowability of the filler metal through the joints; checking the vacuum compatibility of the brazed joint and interdiffusion of base metal and the filler alloy.

2.3 OFHC COPPER

The material that has got the requisite criteria for being used in a RFQ structure is OFHC copper. Oxygen free high conductivity copper (OFHC) has a purity level of more than 99.95%. The rest includes minimal amounts of phosphorous, zinc, cadmium, silver, and tellurium. Oxygen free copper has an oxygen content that has been deliberately minimized. This is accomplished by melting and casting copper cathodes under atmospheres that reduce oxygen and prevent the formation of oxides during casting. Since no deoxidant is introduced into this cast copper, it can absorb some oxygen from the air during long duration heating at high temperatures.

Oxygen free coppers are very ductile. The absence of the copper-copper oxide eutectic improves the cold working properties of these coppers over the oxygen bearing copper. More importantly, from the brazing point of view they can be annealed at high temperatures in reducing temperatures, since they do not get embrittled by hydrogen. The following table gives an outline of the properties of two grades of OFHC copper [Metals Handbook Vol 2].

Table 2.1 Properties of OFHC copper

PHYSICAL PROPERTIES	C10200	C10700
Melting Range	1083°C	1083°C
Density	8.94 gm/cm ³ at 20°C	8.94 gm/cm ³ at 20°C
Specific Gravity	8.94	8.94
Coefficient of Thermal Expansion	17x10 ⁻⁶ /°C (20-300°C)	17.7x10 ⁻⁶ /°C (20-300°C)
Thermal Conductivity	383 - 391 W/m-K	383 W/m-K
Electrical Conductivity (Annealed)	.586 megohm cm at 20°C (101%IACS)	.580 megohm cm at 20°C
Modulus of Elasticity (tension)	12,100 kg/sq mm	12,100 kg/sq mm
Annealing Temperature	375-650°C	475-750°C
Tensile strength	228	235
Machinability	20 free cutting brass =100	20
Hardness	75-90 VHN	45 Rockwell F

2.3.1 Compatibility of OFHC copper for the RFQ structure

The main problem with brazing of copper or copper alloys is that it is done with filler metals that have melting point above 700°C which results in the annealing of the base metals thereby reducing its strength and hardness. OFHC copper (C10200) being highly pure cannot prevent annealing and grain growth. So there is a section of researchers looking for materials with some alloying preferably Aluminum [Valdiveiez et al. 2000] modifications without affecting its primary properties. The obvious choice is to use silver in small amounts of 0.085% (C10700). The slight addition would help prevent grain growth. The silver particles would preferably be sitting in the grain boundaries since it is energetically favored. During grain growth these particle would retard the rate at which the grain boundaries can move out. That is the reason that although we have used C10200 as our material but in our future studies we would definitely like to take a look at C10700.

Apart from the disadvantage stated this material is perfectly suited for the fabrication of RFQ structure. The following reasons would elaborate the situation.

- i) It has very high thermal and electrical conductivity. The primary requirement of the RFQ structure is that the inside surfaces are conducting and smooth. Now the electrical conductivity according to International Annealed Copper Standard is 101%. Moreover the thermal conductivity is among the highest (see table 2.1).
- ii) The machinability of this material is very good. Smoothness was another criterion of the surface which is best achieved in a material like OFHC copper. Almost a mirror like polishing can be achieved as shown in the following figure of a brazed RFQ fabricated at KEK Japan (see figure 2.11).

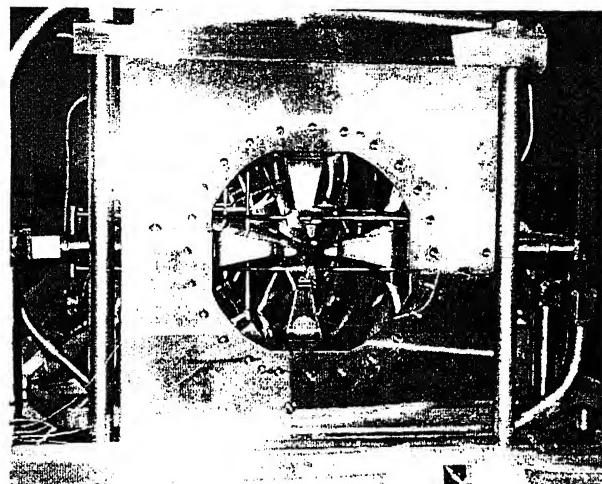


Figure 2.11: A brazed RFQ structure at KEK, Japan

- iii) It has got good vacuum compatibility. Moreover, it has got low vapor pressure.
- iv) Brazing of this material is easy. The range of fillers available in market for brazing copper is very high. As the range increases the ease of brazing increases. Brazing is the easiest joining technique for OFHC copper in comparison to other joining methods.
- v) It has a good mechanical strength. Strength of the material is sufficient for its handling during the whole brazing process.
- vi) It is the popular structure used all over the world. In most of the places where people are working on development of RFQ structure they are using OFHC copper as their material. Although there are some places where OFHC copper is being replaced by GLIDCOP. It is a material consisting of mostly copper with very fine particles of alumina. These fine particles of alumina help in preventing the grain growth during brazing and thereby reduce the mechanical deterioration. But the problem associated in Glidcop is that it makes a huge compromise with the electrical conductivity of the structure. The electrical conductivity comes down to 85% according to IACS standards with addition of 0.1% alumina [R.Valdiveiez et al. 2000].

2.3.2 Earlier work done on OFHC copper

Most of the work done on this material is hardly published. There are very few papers on this material solely. Among those papers the work done by T.Higo et al. of Saitama

University, Japan [T.Higo et al. 2001] is worth mentioning as it has immense application in our future work on accelerator structure. It examines the changes in surface characteristics of copper after each step involved in the processing of the X-Band Accelerator in a scanning electron microscope. The standard fabrication method they had followed is summarized here; 1-rough machining of class 1 OFC, 2-annealing at 500°C, 3-diamond turning about 50m in total, 4-rinsing in acetone ultra-sonic bath, 5-storage in air, 6-ozone-gas included pure water rinsing, 7-storage in air, 8-stacking in clean room, 9-pre-bonding in vacuum furnace, 10-diffusion bonding at 890°C in vacuum furnace, 11-brazing in hydrogen furnace at 1000°C, 12-RF tuning and finally 13-vacuum baking before going into 14-high power feeding. Apart from few steps most of them would be followed during our fabrication process too. The surface morphology is shown below after few of the operations.



Figure 2.12: Surface after HPR at 65kg/cm^2 for 2 hours

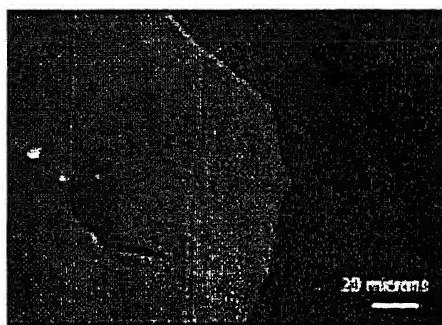


Figure 2.12: Copper surface after brazing heat cycle in hydrogen furnace

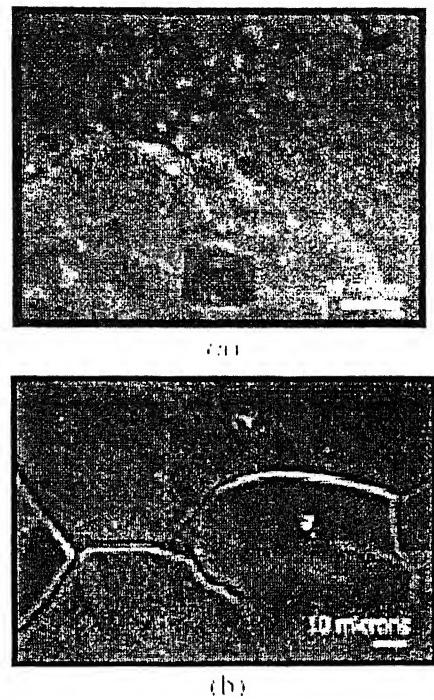


Figure2.12: Copper surface after diffusion bonding heat cycle in vacuum furnace

2.4. BRAZING AND ITS PRINCIPLES

2.4.1. Background

Brazing that developed as an art in ancient times has now evolved into a science through our increased understanding of the behavior of materials in relation to the joining process. In a very general sense brazing is a process for joining materials which relies on the melting flow and solidification of a filler metal to form a leak tight seal, a strong structural bond or both. The uniqueness of the process is that metallurgical bonds are formed during brazing by melting only the filler metal and not the parts to be joined.

The American Welding Society defines brazing as a group of welding processes which produces coalescence of materials by heating them to a suitable temperature and using filler metal having a liquidus temperature above 450°C (840°F) and below the solidus temperature of the base materials. In other words it is a process for joining two or more solid materials in close proximity by introducing a liquid metal (brazing filler metal) that melts above 450°C.

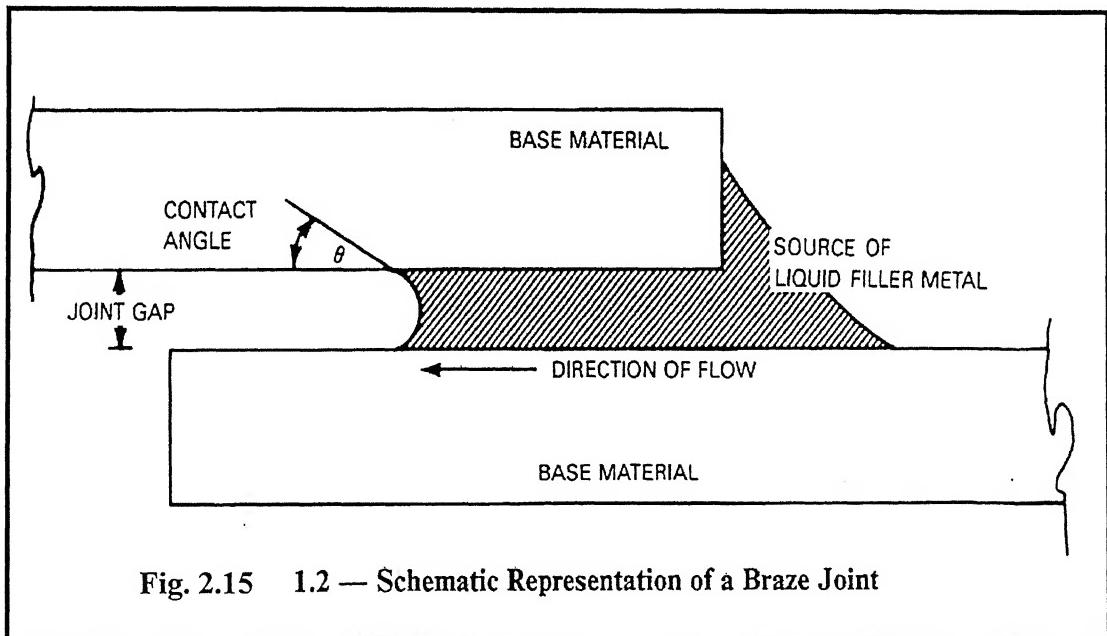


Fig. 2.15 1.2 — Schematic Representation of a Braze Joint

The main differences between brazing and other joining processes are given in the table 2.2.

Table 2.2: Comparison of soldering, brazing, and welding [Metals Handbook Vol 6 1983]

Parameter	Soldering	Brazing	Welding
Joint formed	Mechanical	Metallurgical	Metallurgical
Base metal	Does not melt	Does not melt	Does melt (exceptions exist)
Filler metal melt temperature	< 450°C	> 450°C	> 450°C (optional)

2.4.2. Parameters affecting brazing

The quality of brazed joints depends strongly on the combination of filler and component materials, including surface coating, if any, and also on the processing conditions that are utilized. Hence we require a sound understanding of the metallurgical changes accompanying the sequence of events that occur in making the brazed joints.

Brazing technology has by and large developed through trial and error process. Although theoretical guidelines are there, the basic difficulty is that the real situation is far more complex, as it brings large number of variables. Among the relevant factors are the condition of the surfaces, the temperature gradients that develop during the joining operation, the metallurgical reactions involving the filler and the base metals.

The main aspect of the process nevertheless is the extent of flow of the filler metal inside the joint. This is influenced by the following features:

- Dimensions of the joint.
- Spread characteristics of the filer metal.
- Surface conditions of the components.

The limitation of the theory in accounting for the observed behavior is well illustrated by the classical model of wetting and spreading. This model nevertheless provides useful concepts and insights. It is given a detailed treatment by Harkins [1952], whose main features would be included here.

4.2.1. Surface energy and surface tension

The concepts of surface energy and surface tension would be briefly reviewed. Figure 2.16 provides an oversimplified representation of the atomic structure of solid close to one of its free surfaces. The atom position A in the bulk of the solid has a balanced array of neighboring atoms whereas atom B at the surface is lacking in neighbors above it, apart from the occasional vapour molecule and thus has some unsaturated bonds. Thus the potential energy of the atoms at the free surface, such as B, is higher than the energy of the bulk of the solid, such as A, by the unsaturated bonds. The aggregate of this excess energy that is possessed by atoms in the vicinity of the free surface constitutes the surface free energy of the solid. In the same manner the liquid also possesses surface energy which is manifested in its tendency to draw into drops.

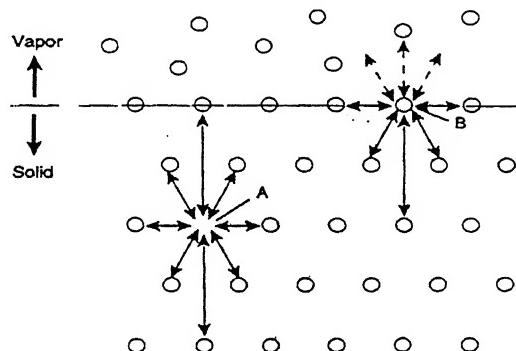


figure 2.16: Simplified diagrams of surface energies. Atom B at the surface has unsaturated bonds and thus a higher energy than atom A. This difference in energy is the origin of γ_{SV}

A surface of a liquid acts like an elastic skin covering the liquid; in other words the surface is in a state of tension. The tensile force (F), known as surface tension (γ), is defined as the force acting at right angles to a line of unit length (L) drawn in the surface.

Consider a liquid film of length L and width W . Apply a force at the barrier AB, as shown in Fig 2.17, parallel to one surface of the film, so as to extend the liquid film a distance

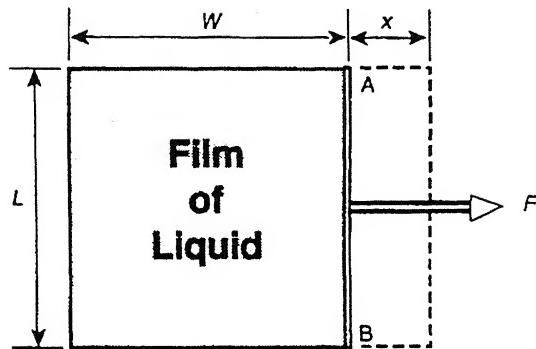


Figure 2.17: Schematic diagram used to explain the relationships between surface energy and surface tension.

The increase in area of the film is xL .

The work done is Fx .

Work done by the liquid in opposing the increase in area under isothermal conditions is $2\gamma xL$.

At a fixed temperature $F/L = 2\gamma$, or F/L for each surface. Thus surface energy is equivalent to surface tension under isothermal conditions [Giles and David, 1993].

2.4.2.2 Wetting and contact angle

According to the classical model of wetting, the liquid will spread over the solid surface until the three surface tensions – between liquid droplet and solid substrate (γ_{SL}), the liquid droplet and the atmosphere (γ_{LV}), and the solid substrate and the atmosphere (γ_{SV}).

The balance of forces is

$$\gamma_{SL} = \gamma_{SV} - \gamma_{LV} \cos\theta \quad (\text{Eq 1.1})$$

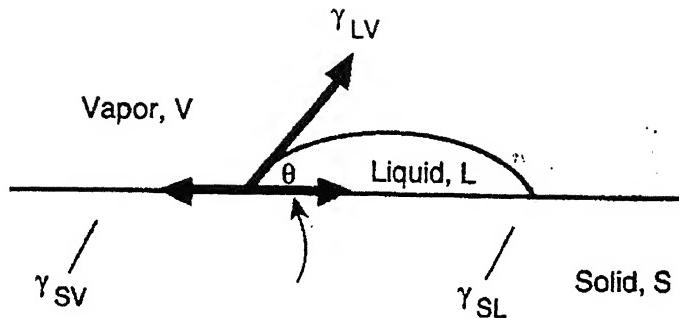


Figure 2.18: Surface tension forces acting when a liquid droplet wets a solid surface, according to the classical model.

Equation (1.1), known as the wetting equation shows that $\theta < 90^\circ$ corresponds to the condition $\gamma_{SV} > \gamma_{SL}$. This imbalance in surface tension provides the driving force for the spreading of liquid over the solid surface and diminution of the unwetted surface area.

Thus contact angle θ provides a measure of the quality of wetting. With θ lying in the range of $90^\circ - 180^\circ$ the liquid droplet would not wet whereas when θ is less than 90° the liquid would spread over an area defined by the contact angle. Clearly the area of spreading increases with decrease in the value of the contact angle. Thus wetting can be improved by

- Increasing γ_{SV}
- Decreasing γ_{SL}
- Decreasing γ_{LV}

These three criteria serve as the basis to which we would always stick during any brazing operation. The implications of the three results are further elucidated in the following paragraphs.

The term γ_{SV} can be maximized for a given solid by cleaning the surfaces. The presence of adsorbed material, such as water vapor, dust, or other nonmetallic surface films on a metal surface, markedly reduces γ_{SV} and correspondingly raises the contact angle. Hence cleaning of the surfaces before brazing is of utmost importance.

The term γ_{SL} is a constant at a fixed temperature for a particular solid-liquid combination, according to the classical model of wetting. Although its value can be changed

by changing the materials system it's never easy. Fortunately, γ_{SL} is highly temperature dependent and decreases with increasing temperature [Schwartz 1987].

The term γ_{LV} is a constant at a fixed temperature and pressure for a particular liquid-vapor combination, but can be varied by altering the composition and pressure of the atmosphere. The spreading is improved by lowering the pressure of the atmosphere. This is one of the reasons of popularity in vacuum based joining processes. The wetting equation for binary metal systems is as follows

$$\cos\theta = 1 + k \left[\frac{T_m^s}{T_m^l} - 1 \right]$$

where k is a constant, equal to 0.3, T_m^s is the melting point of the solid metal, T_m^l is the melting point of the liquid metal. This expression has been verified experimentally [Eustathopoulos and Coudurier 1979].

The study of contact angle gives us an understanding of the capillary effect too. If the contact angle is less than 90° then it would give rise to a positive capillary force that will fill the joint. For a pair of vertical parallel plates, D mm apart and partly immersed in liquid, the capillary force per unit length of joint is equal to $2 \gamma_{LV} \cos\theta$. Under this force the liquid will rise to an equilibrium height h at which the capillary force will exactly balance the hydrostatic force [as shown schematically in Fig 2.19] such that:

$$H = \frac{2 \gamma_{LV} \cos\theta}{\rho g D} \quad \text{Eq 1.2}$$

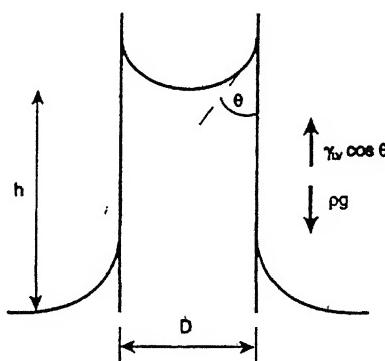


Figure 2.19: Rise of a liquid between two parallel plates

Where ρ is the density of the liquid and g is the acceleration due to gravity. These equations relate to a classical wetting model.

In real brazing process there are deviations from the classical model due the varying degree of chemical reactions between the filler metal and solid surface. This was demonstrated in a study [Schwartz 1987] which showed decrease in the contact angle with time indicating the reactions involved between the substrates and filler metal. Thus in these cases the Gibbs free energy change that occurs on reaction by filler with the substrate is the dominant driving force for wetting.

2.4.2.3 Fluid Flow

According to the classical model, the force that acts to fill the joint gap with liquid is given by Eq 1.3. The liquid will flow into the joint under this force at a rate that is governed by its viscosity. Simple fluid flow theory assumes that

- There is no interaction between the liquid and the solid surfaces with which it is in contact.
- All surfaces are smooth and perfectly clean.
- Flow is laminar, not turbulent.

For a detailed treatment of this subject, the reader is referred to a paper by Milner [1958]. However we shall merely quote the expression for the volume rate of liquid flow, dV/dt , between a pair of horizontal parallel plates, length l , separated by a distance D , under a pressure P per unit area transverse to the plates. The viscosity of the liquid is η .

$$\frac{dV}{dT} = \frac{PD^3}{12\eta l} \quad \text{Eq 1.3}$$

It is assumed that the liquid front will advance at a rate (dl/dt) equal to mean velocity of flow, that is:

$$\frac{dl}{dt} = \left(\frac{1}{D} \right) \left(\frac{dV}{dt} \right) = \frac{PD^2}{12\eta l}$$

From wetting equation 1.1 under isothermal condition the change in surface energy as a unit area of a surface becomes wetted by a liquid is:

$$\gamma_{SL} - \gamma_{SV} = -\gamma_{LV} \cos\theta$$

Therefore, the change in surface energy when the pair of parallel plates becomes wetted is:

$$2l(\gamma_{SL} - \gamma_{SV}) = -2l\gamma_{LV} \cos\theta$$

It follows that the force acting on the liquid to cause it wet the plates is:

$$F = \frac{2l\gamma_{LV} \cos\theta}{l}$$

So that the pressure is:

$$P = \frac{2\gamma_{LV} \cos\theta}{l}$$

And the velocity of flow of the liquid into the space between two parallel surfaces, of separation D, according to this simple model is given by:

$$\frac{dl}{dt} = \frac{\gamma_{LV} D \cos\theta}{6\eta l} \quad \text{Eq 1.4}$$

putting values in this equation shows filling time of the order of 0.01 s. De Gennes [1985] offers a more developed model of the dynamics of liquid spreading, in which the surface energy driving force is opposed by viscous drag and surface irregularities.

2.4.2.4 Filler spreading characteristics

The spreading characteristics of all molten materials are not the same but the spreading characteristics definitely improve with increasing temperature and decreasing the pressure. The main ingredient for better spreading characteristics as has already been mentioned is a perfectly clean surface and which does not get involved with chemical reactions with the liquid filler metal. The wettability index is often used as a criterion for measuring filler spreading characteristics. It was developed by Fedusa and it signifies the area covered by the braze metal times the cosine of the contact angle between the braze and the base metal. Wetting indices greater than 0.05 are indicative of good performance during brazing while greater than 0.10 indicates excellent performance [Chu, 1976].

Eutectic composition alloys are often regarded as having the best spreading characteristics owing to their single melting point. As a result it melts instantly. This is the reason why it is regarded as a better filler metal than its hypo or hyper composition alloys. In case of non-eutectic filler, melting, wetting and spreading commence before the alloy is totally molten, when it tends to be somewhat viscous. Under this condition the movement of

the filler will be relatively sluggish. By the time, the alloy is completely molten the filler metal will partly get alloyed with the substrate, and the driving force for spreading will have been diminished. Moreover, eutectic composition alloys have lower viscosity than adjacent compositions when completely molten. This phenomenon is explained schematically in context of copper-silver brazing filler metal that would be used in our experiments.

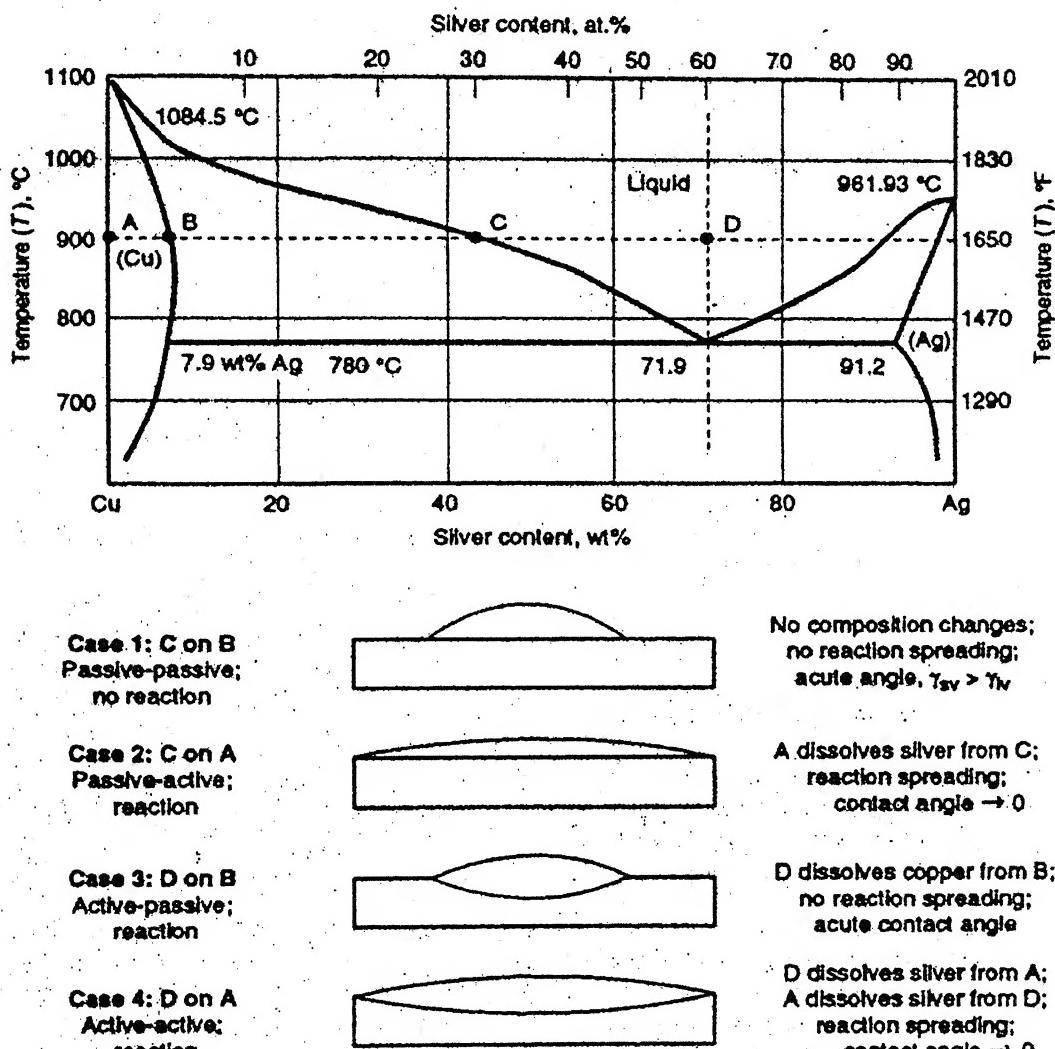


Figure 2.20: Results of a sessile test to analyze spreadability as a function of the solid and liquid composition [Sharps et.al 1981]

The spreading characteristics of braze tend to be less sensitive to composition, because the constituent elements are usually extensively soluble in both the liquid and solid states.

Although high fluidity of a filler metal is a desirable property when it is required to flow into the joint gap of a heated assembly by capillary action, it is not quite so important when the preferred method of applying the filler metal is to sandwich a thin foil preform or layer of paste between the components, which are then joined together in an appropriate heating cycle. This type of spreading is highly detrimental to joint filling, as the filler metal tends to flow out of the joint.

In vacuum or neutral atmosphere, the spreading of filler will tend to be inferior to that obtained in air or in presence of flux or reducing atmosphere. This is to be expected in view of the limited effectiveness of these environments since they are unable to remove the oxide layers.

2.4.2.5 Surface roughness of components

The surface roughness of joint surfaces can have a significant effect on the wetting and spreading behavior of filler metal. The filler metal is drawn into the joint by capillary action. If the surfaces are too smooth then no gap is left between the surfaces and the filler metal may not be able to distribute itself throughout the joint. Thus the resulting voids lower the strength of the joint. Highly fluid filler metals at brazing temperature hardly need any surface roughness but with not so fluid filler metals surface roughness helps to pull the liquid filler metal and distribute it through the joint.

To assure adequate flow through the joint, particularly when the clearance is zero or press fit, the faying surface should be roughened preferably with a metallic grit compatible with the base metal. Surfaces that are too rough will also lower the strength of the joint because only high points will get brazed.

Surface roughness reduces the effective contact angle θ^* , where θ^* is related to θ , the contact angle for a perfectly flat surface, through the relation

$$\cos \theta^* = r \cos \theta$$

where $r = (\text{actual area of rough surface}) / (\text{plan area})$

At the same time, by producing fine channels, the texturing may increase the capillary force acting between the filler and the component surfaces. A directionally oriented surface texture promotes preferential flow parallel to the channeling [Nicholas and Crispin 1986]. Attempts have also been made to improve joint filling by introducing capillary enhancers to

the joint gap. Such enhancers include finely divided powders and fine meshes that are wetted by the filler but that are effectively inert. This type of approach has been explored by researchers, [Nicholas and Crispin 1986] however, this and has not been found to radically improve joint filling.

.4.2.6 Dissolution of parent materials by molten fillers

It is frequently observed that a filler metal will continue to spread beyond an initially wetted surface area over an extended period of time (>10 s), which would not be expected from classical fluid theory. Clearly, classical expressions for fluid flow, Eq 1.4, do not strictly apply in such cases. Indeed, this type of flow can usually be associated with solid-liquid interfacial reactions, which are neglected in the model described in Milner's paper [1958]. In cases where the flow is sluggish due to the reaction between the base metal and the filler the increase in temperature is not going to solve the problem. Dissolution of the substrate and resulting growth of the intermetallic compounds both follow Arrhenius-type relationships, represented by the expression:

$$\text{Rate} = \exp\left(\frac{q}{kT}\right)$$

Where q is an activation energy that characterizes the reaction taking place at temperature T (in Kelvin), which is proportional to the melting point T_m , [Birchenall 1959] and k is the Boltzmann constant. The rate of reaction will therefore be depended on the homologous temperature defined as the ration of T/T_m and will be more pronounced on soldered joint than brazed joints [Frear et al. 1991]. Therefore the alternative is not to increase the temperature but to change the materials system.

Interfacial reactions are important, not only in determining the flow characteristics but also the properties of the joints. When a molten filler metal wets the parent materials, there is normally some intersolubility between them. It is usually manifested as dissolution of the surfaces of the parent materials in the joint region and the formation of new phases at either the interface between the parent material and the molten filler metal or within the filler metal itself when it solidifies.

The following expression describes the rate of dissolution of a solid metal [Weeks et.al 1990]:

$$\frac{dC}{dT} = \frac{KA(C_s - C)}{V} \quad \text{Eq 1.5}$$

where C is the instantaneous concentration of the dissolved metal in the melt, C_s is the concentration limit of the dissolved metal in the melt at the temperature of interest, t is the time, K is the dissociation rate constant, A is the wetted surface area, and V is the volume of the melt. This equation is known as both the Nerst-Shchukarev and Berthoud equation. In the integral form, Eq 1.5 becomes:

$$C = C_s \left[1 - \exp\left(\frac{-KAt}{V}\right) \right] \quad \text{Eq 1.6}$$

Equation 1.6 reflects the fact that, in general, the concentration of dissolved metal in the molten filler increases in an exponential manner with respect to time. That is the dissolution rate is very fast initially, but then slows as the concentration of the dissolved parent material tends towards its saturation limit as shown in Fig 2.21. Substituting measured values into Eq 1.6 shows that, for a soldered or brazed joint of typical geometry, the equilibrium condition is reached within seconds at the process temperature. Thus it is possible to use an equilibrium phase diagram to predict the change in the composition of the filler metal that will occur in typical joining operation and the associated depth of erosion of the joint surfaces. Sometimes the reactions lead to the formation of intermetallics which effectively prevents further dissolution of the parent metal since diffusion through solid is two orders of magnitude slower than solid-liquid reactions [Pfahl et al., 1975]. Brazing also leads to limited dissolution and plastic deformation of the base metal [Liberman, 1998].

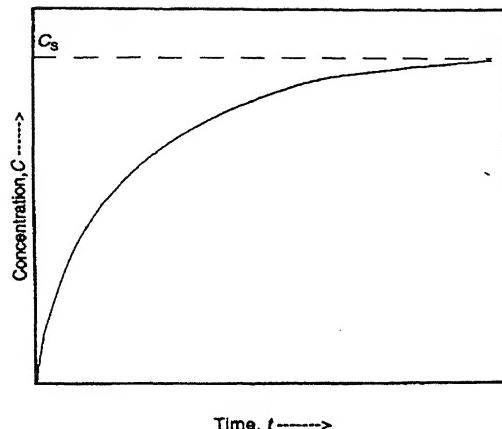


Figure 2.21: The concentration of solid metal in a liquid metal wetted by it changes in an inverse exponential manner with respect to time and is limited by the saturation concentration of the solid constituent in the liquid at that temperature.

Moreover some recent work in Japan [ISO 9453, 1990] and United states [ISO 9454-1, 1990] has shown the effects of base metal and filler metal reactions on the wetting of Si_3N_4 and titanium added active filler metal vary with reaction layer thickness and morphology as well as with the composition of the reaction layer.

2.4.2.7 Significance of the joint gap

Clearance or joint gap is the distance between the faying surfaces of the joint. The clearance between the members of similar metals is easily maintained in assemblies where the parts are pressed or require shrink fit. When designing with dissimilar metals, the clearance must be calculated for the brazing temperature. The joint gap at the process temperature influences both the joint filling and the mechanical properties of the resulting joint. The mechanical properties include all types of loading like static, fatigue, impact etc. joint clearance has several effects on mechanical performance and includes

- I) The purely mechanical effect of restraint to plastic flow of the filler metal offered by the greater strength of the base metal.
- II) The possibility of flux entrapment.
- III) The possibility of voids.
- IV) The relationship between the joint clearance and the capillary force which accounts for the filler metal distribution.

To assure optimum flow into the joint it may be necessary to use spacer wires, shims, prick punch marks, grit blasting etc. The importance of joint gap study is the most relevant part in brazing because the joining is totally dependant on the capillary flow of the molten filler. So it is imperative to understand the parameters that could have huge influence on the election of the clearance.

2.4.2.7.1 Effect of fluxes and protective atmospheres

Fluxes are added to prevent the formation of oxides and other undesirable elements during the brazing cycle. These fluxes melt at a temperature below the melting point of brazing filler metal and must flow into the joint to prepare it for the filler metal. The filler metal on melting must force out the flux as the filler metal is drawn into the joint by capillary action. When the clearance is too small, the flux will be entrapped in the joint so firmly that the displacement by the filler metal might be difficult. Thus joint defects may be produced. When the clearance is too large, the liquid filler metal will travel around the pockets of flux, thus giving rise to excessive flux inclusions.

Protective atmospheres have the same function as the fluxes. The protective atmospheres in general require low clearance to obtain the optimum strength, so that the load carrying capacity of the joint can be higher. In general, when the protective atmosphere is used the filler metal flows out if the clearance is more than $80\mu\text{m}$ (0.08mm). Large clearance joints require filler metals having wide range of melting point. The purity of the atmosphere is also found to affect the clearance value. Higher purity atmospheres require low clearance.

2.4.2.7.2 Effect of surface finish on clearance

The effect due to the surface finish has already been discussed in section 2.4.2.5.

2.4.2.7.3 Effect of base metal-filler metal interaction

This has also been discussed in section 2.4.2.6. The only point here to be noted is that the clearance increases as the joint length increases to prevent freezing of filler metal in the joint as it picks up more base metal.

4.2.7.4 Effect of base metal on clearance

The chemistry of base metal often includes one or more elements in varying quantities whose oxides are not easily dissociated in a specific atmosphere or by a specific flux. Since the metal-metal oxide dissociation of a given base metal by a specific flux or protective atmosphere is dependant on many factors, it is important to match the proper clearance with the brazing process and flux or atmosphere.

Table 2.3: Recommended joint clearance at brazing temperature [Brazing Handbook, 1991]

Filler metal AWS Classification	Joint Clearance, μm
BA1Si group	0-50 for furnace brazing in vacuum atmosphere
	50-220 for length of lap less than 0.62cm
	220-290 for length of lap greater than 0.62cm
BCuP group	25-140 no flux and for flux joint length less than 2.5cm
	200-425 no flux and for flux brazing joint length > 2.5cm
BAg group	50-140 flux brazing
	0-50 for atmosphere brazing
BAu group	Same as above
BCu group	0-50 atmosphere brazing
BCuZn group	50-140 flux brazing
BMg group	100-300 flux brazing
BNi group	0-50 free flowing type, atmosphere brazing

The degree of mutual solubility of the base material and the filler metal will also have an effect on the optimum clearance. When the solubility is high and the clearance is low, the pick up of the base metal as the filler metal flows through the joint will progressively increase the melting temperature of the filler metal in the joint. Thus, the filler metal can solidify in the joint without completely filling the joint.

The co-efficient of thermal expansion will have an effect on the joint clearance at brazing temperature when the base materials are dissimilar.

2.4.2.7.5 Effect of brazing filler metal on clearance

Filler metals have a broad spectrum of flowability and viscosity at various brazing temperatures and conditions. Some flow freely and others are sluggish. The free flowing filler metals require low clearance than the sluggish fillers. Filler metals that have a single melting point, such as pure metals, or eutectic melting filler metals and self fluxing filler metals, will usually be free flowing, particularly when there is no or very little interaction with the base metal.

Variations in the fluxes and the atmosphere can enhance the free flowing qualities of the filler metals or can result in no flow at all as fluxes may be a poor choice for a given application. Thus it is essential to choose the proper flux or atmosphere.

2.4.2.7.6 Effect of joint length and geometry on Clearance

As the joint length increases, the clearance must also increase, particularly when there is interaction between the filler metal and the base metal. As the filler metal is drawn into a long joint it may pick up enough base metals to freeze before it reaches the other end. The more the interaction between the base and the filler metal the larger should be the clearance. This is one of the most important reasons as to why the joint length should be made as short as possible to optimize the joint strength.

2.4.2.7.7 Effect of dissimilar base metals

When designing a joint where dissimilar base materials are involved, it will be necessary to calculate the joint clearance at the brazing temperature. The thermal coefficient of expansion for each member and the brazing temperature must be taken into consideration to assure the desired mechanical strength in the final joint. With high differential thermal expansion between two parts, the brazing filler metal must be strong enough to resist fracture and at least one of the base materials, filler metal, or an added layer of ductile base metal must yield during cooling. It is important to remember that some residual stress will remain in the final braze as a result of joining at the brazing temperature and subsequently cooling to room temperature. Thermal cycling of such a braze will reduce the life of the joint.

2.4.3. Functional and design requirements

All brazed joints used in manufactured products must remain solid in service and retain their associated components in fixed positions when subjected to stress. These requirements are usually satisfied by suitable designs of the geometry and the metallurgy of the joint, but there are also other aspects to consider. Several factors that affect the functional integrity of the joints are discussed below.

2.4.3.1 Metallurgical stability

It is important that the brazing temperature does not increase the recrystallization and grain growth temperature of the base materials. Although at times it might be necessary. The phases that form on solidification should be stable. Else the joint can affect the integrity of the structure. Since most of the fillers are softer than the base materials hence the properties of the joint are limited by those of the filler.

2.4.3.2 Mechanical integrity

The durability of the engineering and consumer products often depends on joints maintaining their mechanical integrity for the entire service period. The following factors affect its mechanical integrity

- The mechanical properties of the bulk filler metal.
- The joint geometry- namely area, length, width and shape.
- The mechanical properties of any new phases formed in the joint by reaction between the filler and the components, either during the joining operation or subsequently in service.
- The number, size, shape, and distribution of voids within the joint.
- The quality of fillets formed between the filler and the surface of the components at the edge of the joint.

2.4.3.3 Environmental durability

Joints are normally expected to be robust in relation to the service environment. This most commonly involves exposure to corrosive gases, including sulfur-di-oxide and other

constituents of polluted atmosphere; to moisture perhaps laden with salt; to variable temperature. The corrosion and stress-corrosion characteristics of the joint gains relevance. Each situation should be determined empirically. In other words there is interdependence between environmental stability and microstructural stability. Appropriate choice of materials combination used should enable these changes to be constrained within predictable and acceptable limits.

2.4.3.4 Electrical and thermal conductivity

In certain applications the brazed joint should be able to maintain the electrical and thermal continuity. It is here that it is required that the filler metal should also have almost the same values of electrical and thermal conductivity as that of the base materials. Moreover the brazed joint must be of almost zero thickness. It is only possible with extreme caution in designing of the joint, placement of the filler metal and good fluidity of the filler metal.

2.4.4. Processing aspects

The most important aspect of the brazing process is the practicality of the process involved. Hence a step by step formulation of the brazing procedure is being listed with brief explanation of each point.

2.4.4.1 Jigging of the components

The components being joined normally must be held in the required configuration until the filler metal has solidified. It is mentioned that the best way of placing any components is self jigging. Even if the components can be preplaced without a fixture, the use of some form of jig is still beneficial to ensure that the components are not disturbed by capillary forces originating from the molten filler metal.

The jig if required should fulfill the following qualities. It should be nonporous material to prevent contamination of the air surrounding the workpiece. It should neither be wettable by the filler metal. This is the reason why graphite is often favored as a jigging material. Moreover graphite helps in mopping up oxygen. Sometimes the jigs are also used to apply a controlled pressure.

2.4.4.2 Form of the filler metal

Filler metals are available in many different forms. These include configurations that can be normally produced from an ingot by mechanical working- for example, wire, rings, and foils. The development of rapid solidification technique has led to the availability of foils and wire of joining that are inherently brittle. Fig 1.6 shows few of the forms of filler metals that are easily available in the market. They are also available in the form of paste capable of being screen printed onto substrate or applied to a workpiece.

In a more specialized joining process braze can be deposited by electroplating and by vapor deposition techniques such as evaporation.

But the use of some form of preplaced filler metal has a number of advantages. Most particularly, because thickness and the area of the filler metal are predetermined, the volume of molten filler metal may be carefully controlled. This can help contribute to a well-defined and reproducible process that gives consistent results.

2.4.4.3 Heating methods

Heat must be supplied to the joints to raise the temperature of the joint surfaces and the filler metal above the melting point of the filler. The components also need to be heated up to prevent balling up of the filler metal. Hence it is best suited to heat the filler metal via the components to be joined. The available methods of heating are local heating in which only those parts at the vicinity of the joint would be heated and diffused heating where the entire temperature of the assembly is raised at the required level.

The former technique includes torch brazing, resistive heating of the joints, induction heating, and laser heating. The other mode of supplying heat includes the use of furnaces which can be resistive or optical mode.

2.4.4.4 Temperature measurement

It is a very important measurement during brazing operation. Usually the actual brazing time varies from few seconds to over an hour depending on the mass of the components. Hence the temperature at which the brazing is done assumes criticality. Thus measurement of the temperature has to be correct. Therefore the instrument by which the temperature is measured has to be calibrated in accordance with the heating source using a

standard element having a particular melting point. Correct temperature measurement requires good thermal contact between the thermocouple and the object being monitored. This presents a problem in vacuum furnaces. The use of pyrometers offers an advantage since they require no contact. Traditional pyrometers are designed to measure temperature above 750°C.

2.4.4.5 Joining atmosphere

For a molten filler metal to wet and bond to a surface, the latter must be free from nonmetallic surface films. Although this condition should be met at the beginning of the heating cycle, by prescribed cleaning treatments, significant oxidation will generally occur if the components are heated in air. Steps must therefore be taken to either prevent the oxide film as fast as it forms. The approach largely depends upon the type of atmosphere surrounding the workpiece [Bannos 1984]. Brazing is conducted in the following three types of atmosphere.

I) Oxidizing atmosphere

When brazing is done in open atmosphere as in torch brazing then we term the atmosphere as oxidizing. But in this type of atmosphere there is every chance that oxidation of the surface might occur during the brazing cycle hence use of flux covering over the joining surfaces is recommended. Materials like gold, platinum and noble metals do not get oxidized in air and hence can be brazed in an oxidizing atmosphere.

II) Inert atmosphere

From a practical point of view, an atmosphere is either oxidizing or reducing. This is because it is not possible to remove and then totally exclude oxygen from the workpiece, except perhaps under rigorous laboratory conditions. Thus an inert atmosphere must be defined in relation to the residual level of oxygen. Typical inert atmosphere includes nitrogen, argon, and vacuum.

III) Reducing atmosphere

It is an atmosphere that is capable of chemically removing surface contamination on metals. Gases that provide reducing conditions are generally proprietary mixtures that liberate halogen radicals. For a few metals hydrogen is also

preferred. Hydrogen is a difficult gas to dry, and the water vapor present can present serious problem. Moreover hydrogen can embrittle few metals at high temperature.

2.4.4.6 Cleaning treatments

The surfaces of the components to be joined and the filler metal preforms must be free from nonmetallic films, such as organic residues and metal oxides, to enable the molten filler metal to wet and alloy with the underlying metal. Fluxes are often capable of removing surface oxides, provided they are reasonably thin. Organic films can be removed with solvents, which obviously should not react with the underlying materials. Thick oxides and other nonmetallic surface layers can be removed chemically. However mechanical cleaning is preferred since chemicals tend to leave residues, which then also have to be removed. Dry mechanical abrasion exposes a fresh metal surface. The roughness of the abraded surfaces can be readily controlled, and this can be used to advantage in promoting the spreading of the molten filler metal.

2.4.4.7 Heating cycle of the joining operation

The prepared samples possibly mounted in jigs, are joined by applying heat. The heating cycle involves four important processing parameters: the heating rate, the peak bonding temperature, the holding time above the melting point of the filler, and the cooling rate. In general a fast heating rate is prescribed to limit the reactions that can occur below the prescribed bonding temperature. However the maximum heating rate is normally constrained by the adverse thermal gradient that develop in the component. A better practice is to heat the sample to a preset temperature that is just below the melting point of the filler metal and then hold at this temperature for sufficient time to equilibrate the temperature gradient through out the body of the sample. Following this cycle the sample should be rapidly heated to the bonding temperature. The bonding temperature should be such that the filler metal is guaranteed to melt, but at the same time it should not be so high that it degrades through the loss of its constituents by reaction with the furnace atmosphere. The minimum time that the assembly is held for should be enough to ensure that the filler has melted over the entire area of the joint. The cooling time is seldom controlled; rather it is a function of the mass of the assembly and jig. Occasionally one or more dwell stage is also required during brazing

CHAPTER 3**EXPERIMENTAL PROCEDURE****3.1 STUDY OF DEGRADATION OF MECHANICAL PROPERTIES OF OFHC COPPER AFTER BRAZING**

The material required to fabricate the radio frequency quadrupole was chosen as oxygen free high conductivity copper. This material has found worldwide recognition due to its many advantages which has been already mentioned in the previous sections. The fabrication of the whole structure requires joining of similar parts to one another by the brazing technique. The heat required for brazing can be best supplied by a hydrogen furnace. Hydrogen furnace offers some basic advantages like removal of oxide films during brazing, uniform heating of the whole structure which would help prevent build up of stresses due to thermal gradient and good control over temperature at the joints, thereby allowing simultaneous brazing of all the joints.

To braze the joints successfully the main requirement is a good brazing filler alloy that has good fluidity at bonding temperature and good compatibility with the base metal. The brazing filler alloy that can be used for preparing good brazing joints were then identified which are mentioned below [Brazing Handbook, AWS]

1. BAg-6 (49.0-51.0% Ag, 0.001% Zn, Cd, 0.002% Pb, P, 0.005% C, remainder Cu)

The range of temperature at which brazing can be done using this filler metal is 774-871°C. It is a widely used filler metal in brazing industry, joining brass parts, in the dairy and food industries where the use of cadmium is prohibited. This filler metal has a broad melting range and is not so free flowing as BAg-1 or BAg-2; it is good filler for bridging gaps or forming fillets.

2. BAg-8 (71.0-73.0% Ag, 0.001% Zn, Cd, 0.002% Pb, P, 0.005% C, remainder Cu)

The range of temperature at which brazing can be done using this filler metal is 779-899°C. This is a filler metal that is suitable for use in controlled-atmosphere furnace brazing (including vacuum brazing) without the use of a paste flux. It is usually used on copper and copper alloys. When molten BAg-8 is very fluid and may flow out of the work piece during some brazing applications. It is also used on stainless steel, nickel base alloys.

and carbon steel, although it's wetting action on these metals is slow. But increasing the temperatures will improve flow and wetting.

1. BVAg-29 (60.5-62.5% Ag, 14-15% In, 0.001% Zn, Cd, 0.002% Pb, P, 0.005% C, remainder Cu)

The range of temperature at which brazing can be done using this filler metal is 707-788°C. It is the lowest melting filler metal of the low vapor pressure alloys. The indium content of this filler metal improves wetting action over compared to BAg-8.

2. BAu-2 (79.5-80.5% Au, 0.001% Zn, Cd, 0.002% Pb, P, 0.005% C, remainder Cu)

The range of temperature at which brazing can be done using this filler metal is 891-1010°C. It is a very high temperature brazing alloy. The main purpose of using this alloy would be high fluidity and application in step brazing technology.

It is clear by now that the temperatures required for brazing copper whose melting point is 1083°C is on the higher side. Thus the brazing of copper at high temperatures leads to simultaneous recrystallization and grain growth of OFHC copper.

Now we know that with grain growth the yield and tensile strength get reduced deteriorating the overall mechanical properties. Further the hardness would also decrease as we go for higher temperature brazing alloy. So the first part of my experiments involves the study of this deterioration of the mechanical properties during the brazing experiments.

To measure the decrease in the mechanical properties the OFHC copper samples were kept in a condition simulating the brazing process and then measurement of the tensile, yield, hardness and study of grain growth was done.

3.1.1 Steps in experimental work

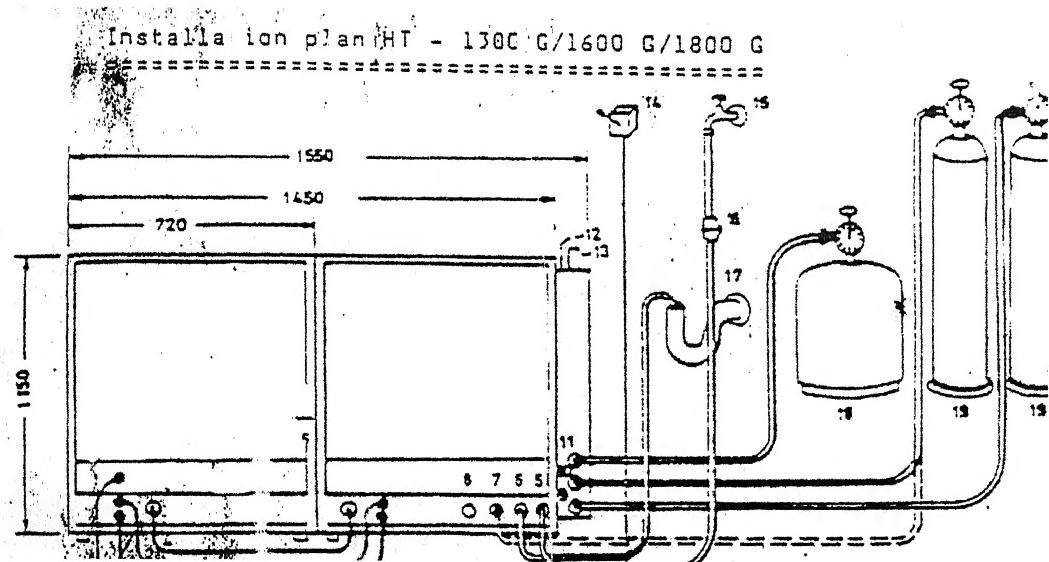
I) Sample preparation: The study that has to be done is measurement of decrease in tensile strength, yield strength, and hardness values. Hence tensile specimens of a given dimension, according to the ASTM standards (ASTM E8) were machined in lathe and prepared. Two sets of tensile samples having dimensions of 50mm gauge length and 12.5mm radius, 70mm gauge length and 14mm radius were machined. Along with this another sample of 5cm x 5cm x 5cm was machined. This sample was used to measure the hardness change and the grain growth due to the heating. The tensile sample preparation

was mostly done on a lathe with an error of 0.1-0.5 mm. few of the bigger samples were machined in a CNC milling machine with a machine accuracy of 1 μ m. The machining was done at two different places, at Centre of Advanced Technology, Indore and at Indian Institute of Technology, Kanpur.

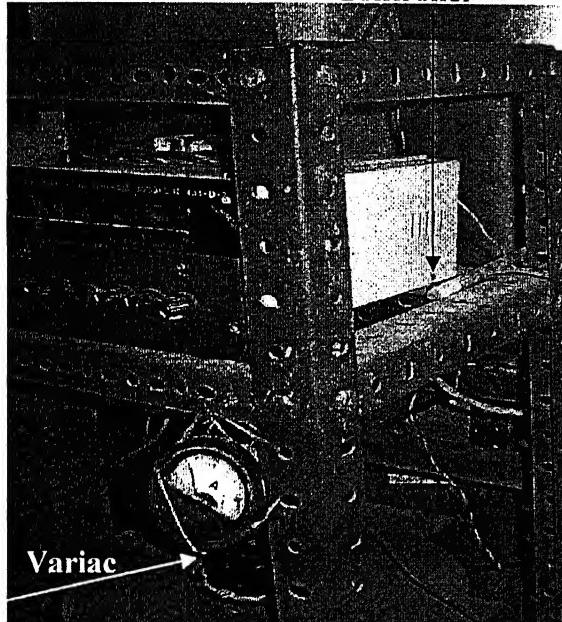
ii) Cleaning: After the sample preparation it was cleaned in a special soap solution called Exalin (Merck Company) and then dried in ethanol solution to make it grease and dirt free. This step was done to ensure that the atmosphere inside the furnace, during the heating process does not get contaminated. Moreover this chemical is eco-friendly and bio-degradable.

iii) Gas flushing: The sample after cleaning was loaded in the furnace. After loading it in the furnace nitrogen flushing was done to remove the total residual oxygen inside the furnace. The time of flushing varied depending on the size of the furnace. Two different types of furnaces were used in this experiment. The larger furnace had a dimension of 1m x 30cm (diameter). The total time of flushing was for 30 minutes at a rate of 90cfh. The smaller furnace was a cylindrical one with a diameter of 25.4 mm and length of around 80 cm. the flushing time was around 10 minutes. A schematic of the larger furnace and a photograph of the smaller furnace used are both shown in Fig 3.1 in next page. Argon gas can also be used in place of nitrogen. Both being heavier than oxygen can easily replace them from the inside of the furnace.

The hydrogen gas flushing was then switched on while the rate of nitrogen flushing was reduced to zero in steps. As the hydrogen started coming out of the furnace through the outlet it was ignited with a burner. It takes some time before hydrogen is ignited at the outlet since it has to replace the whole of nitrogen gas inside which is a heavier gas. Hydrogen gas is a very difficult gas to keep dry and hence it is of importance to mention the dew point. The dew point value for the larger furnace is only available. The value of the dew point is -50°C although this value keeps changing with the temperature, inside the furnace. In fact the dew point keeps increasing with temperature.



Controller



Furnace

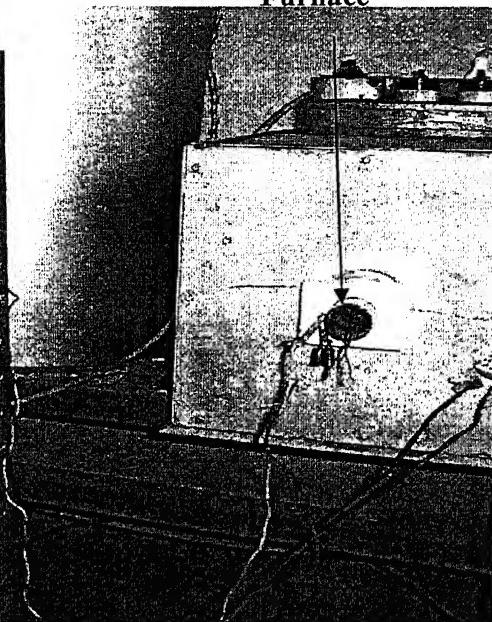


Figure 3.1: It shows the furnaces used for the experiments. The top one is the larger Furnace (CAT Indore) while the bottom one is the smaller furnace (IIT Kanpur)

iv) Heating cycle: After hydrogen gas started burning at the outlet, the heating of the furnace was started. Heating involved a continuous increase of the temperature at almost a specific rate and then holding the samples at a peak temperature for a scheduled time interval. The peak temperature was determined using the range of temperature required for brazing of the sample with a particular brazing filler metal. Usually the time required for brazing at the peak temperature was not more than 5 minutes. Depending on the mass of the assembly we had to introduce a soaking period during the actual brazing process. Since we were just concerned with the deterioration of the mechanical properties, the soaking period was not considered. One more reason why this soaking period was removed is that copper being a highly thermally conducting material would not develop thermal gradient at all. The thermal cycle involved in the process is shown in Fig 3.2 and 3.3. The temperatures at which the tests were done are 800°C, 830°C, 860°C, 950°C, and 1000°C.

v) Cooling cycle: The cooling was started after keeping the sample at the peak temperature for 5 minutes. The heat supply was switched off, but the hydrogen gas flushing continued. Initially cooling was very fast at a specific rate but as it approached the arrest point of the furnace the cooling rate decreased sharply and the actual time required to cool the sample to the room temperature increased thereby decreasing the overall cooling rate. The arrest point of the furnaces that has been used for experimental purpose was around 170°C – 270°C.

vi) Gas Flushing: Before taking out the sample the hydrogen flushing was stopped and nitrogen was started again to remove the residual hydrogen. Nitrogen flushing, again depending on the size of the furnace was done for 15 minutes for the bigger furnace and for 5 minutes for the smaller furnace. After that nitrogen flushing was stopped and the samples were taken out at around 50°C- 70°C.

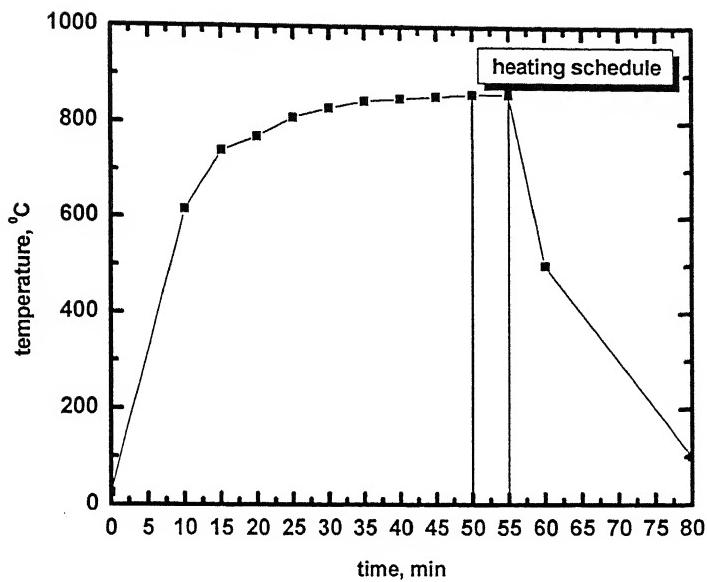


Figure 3.2: The heating cycle of larger cylindrical furnace at CAT Indore

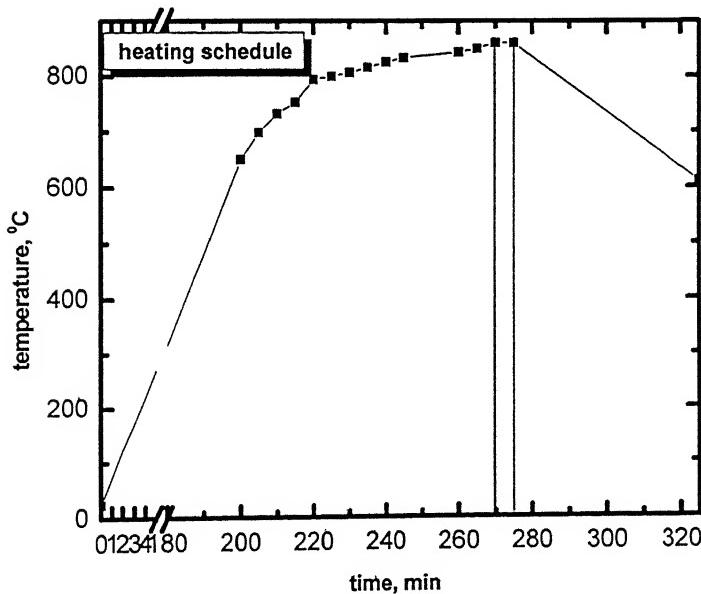


Figure 3.3: The heating cycle of smaller cylindrical furnace at IIT Kanpur

3.1.2 Evaluation processes of the samples

After the thermal cycle was over the following tests were done on the samples to study the deterioration of the mechanical properties.

- Tensile strength evaluations of those samples were done in a MTS machine which had a gauge length of 50mm and gauge diameter of 12mm. Two different sets of experiments were done to evaluate the tensile strength. The first set had a heating rate of 700°C /hr while the second had a rate of 150°C /hr.
- Micro hardness testing was done on 160mhp microhardness testing machine with a load of 20gm (0.1962N).
- Metallographic studies were done using an image analyzer at various magnifications of which the results of 500X magnification were compared. Metallographic examination is often regarded as involving little more than straight forward observation. Great care must be taken to avoid introducing artifacts of sample preparation else an unrepresentative microstructure is seen [Bjerrgaard et al. 1992].

3.2 STUDY OF THE BRAZING PROCESS OF OFHC COPPER

The study of the brazing process involved the characterization of the process with main emphasis on the fundamental procedure to obtain a good brazing joint. It has been already mentioned that the whole RFQ structure is to be fabricated of OFHC copper by brazing procedure. Hence a greater understanding of the process has to be developed on the basis of some small samples which can be later applied to prototype building and lastly the manufacturing of the actual RFQ structure. So the main aim of the study was to develop a database to fall upon during the actual brazing of the sample structures. In the light of the previous experiments and their results it was concluded that the best brazing material suitable for the brazing procedure was BAg-8. Moreover it was also mentioned in the literature survey that the most common material for RFQ fabrication was the copper–silver eutectic owing to its good bonding and flow properties at the brazing temperature, although the use of some other filler alloys has also been reported. The other most common material is copper-gold-nickel filler alloy.

Many experiments were done on samples of different configurations by varying the heating rate, cooling rate, soaking temperature, soaking time, brazing temperature, and surface roughness. But the essential steps involved in all experiments remained similar. The main steps involved in the brazing process are being listed below. The steps are almost similar to the earlier experiments except some minor differences due to consideration of parameters like soaking temperature and time, brazing temperature, and cleaning procedure.

3.2.1 Steps in experimental work

I) Sample preparation: The samples prepared were of different configurations and shapes to study the flow of the braze filler metal through the joint. The different sets of samples that were prepared are listed as follows.

- Three pairs of small cubic samples.
- Four pairs of cylindrical samples which would be made into tensile samples after brazing.
- A small cylindrical sample which after brazing would be used for scanning electron microscopy study.
- One sample for testing the vacuum tightness of the joints.

- Four shear testing samples.

The tensile samples, vacuum testing samples, and one pair of small cylindrical sample were machined in a lathe having an accuracy upto 0.1mm. The shear testing samples were prepared in a milling machine with an accuracy of 0.01mm. The small cubic samples which had varying dimensions were cut out from the leftover pieces with a hacksaw.

ii) Roughing up of the faying surfaces: Prior to cleaning of the samples the surfaces which are to be joined were roughened up using emery paper with a silicon carbide grit size of 800. This is essential since a flow of molten brazing filler metal is difficult in a totally smooth surface. Hence a uniform degree of roughness is introduced although the uniformity can be best supplied by a milling machine.

iii) Cleaning: Brazing of any joint is as good as its cleaning process prior to loading it in the furnace. This point has been repeatedly stressed in the ‘Brazing and its principles’ section. The surfaces of the sample to be brazed, depending on its material characteristics, would have oxide films as well as dust and grease due to the machining of the samples. Inability to remove the dirt and grease would ultimately lead to a joint having voids and thereby less mechanical strength. Therefore a three step cleaning process was followed. Initial cleaning of samples was done by a biodegradable soap solution called Exalin (Merck Company). It is a mixture of soap solution with potassium dichromate and sulfuric acid. Then it was rinsed thoroughly in clean water. After that it was dipped in strong HNO_3 solution for few seconds and then further rinsed in flowing water. It was then dried in methanol solution and kept in a piece of clean tissue paper. The next step was obviously loading of the sample in the furnace. While loading care was taken that the samples were not touched by hand. Hence a fork was used to lift the samples and place it in the furnace.

iv) Loading of the assembly: The samples to be joined were made into an assembly prior to loading in a furnace. The assembly was preferably prepared without any external jigs. Self jigging is the best form of assembly that can be loaded. It was preferred that the joint to be brazed should be in a horizontal position with the filler metal flowing into the joint. In contrast vertical joints are difficult to braze. Therefore while brazing, the sample was loaded on a solid base parallel to the ground, else the

flow of molten filler metal would also be governed by the external gravity effect. In other words the joint should never be in an inclined position. Sometimes jiggling if necessary, was done preferably by graphite blocks. Mild steel holders were once used for holding the samples which should never be done as it has a very high degassing rate. This leads to degradation of the furnace atmosphere thereby producing low quality brazed joint. Apart from jiggling the assembly part also holds the filler metal. Depending on the type of joints to be brazed the filler metal was either in the form of foil or wire. The thickness of the foil was 250 μm -300 μm and the diameter of the wire was 800 μm .

v) Gas flushing: As mentioned before, the gas flushing routine was followed to prevent formation of oxide films on the joint surfaces. It was done to improve the quality of the joint strength. After loading the sample in the furnace nitrogen flushing was done to remove the total residual oxygen inside the furnace. The time of flushing varied depending on the size of the furnace. Argon gas can also be used in place of nitrogen. Both being heavier than oxygen can easily replace them from the inside of the furnace.

Hydrogen gas flushing was then switched on while the rate of nitrogen flushing was reduced to zero in steps. As the hydrogen started coming out of the furnace through the outlet it was ignited with a burner. After hydrogen started burning at the outlet, heating of the furnace was started at constant rate.

vi) Heating schedule: The heating schedule that was usually followed, depending on the mass of the samples to be brazed, is shown in Fig 3.4. It has been already mentioned that the brazing filler metal used was BAg-8, which is a copper-silver eutectic having a melting point of 779°C. Manufacturers of this filler have placed an error of $\pm 1.5\%$. Hence the soaking temperature was identified as 800°C. The soaking time varied depending on the mass of the assembly to be brazed. But normally it was between 20-30 minutes. After the soaking period the temperature was further increased in steps of 10°C to a maximum bonding temperature varying from 810°C-840°C. The sample was again kept at the maximum bonding temperature for 5 minutes. Then the heating was stopped and cooling started.

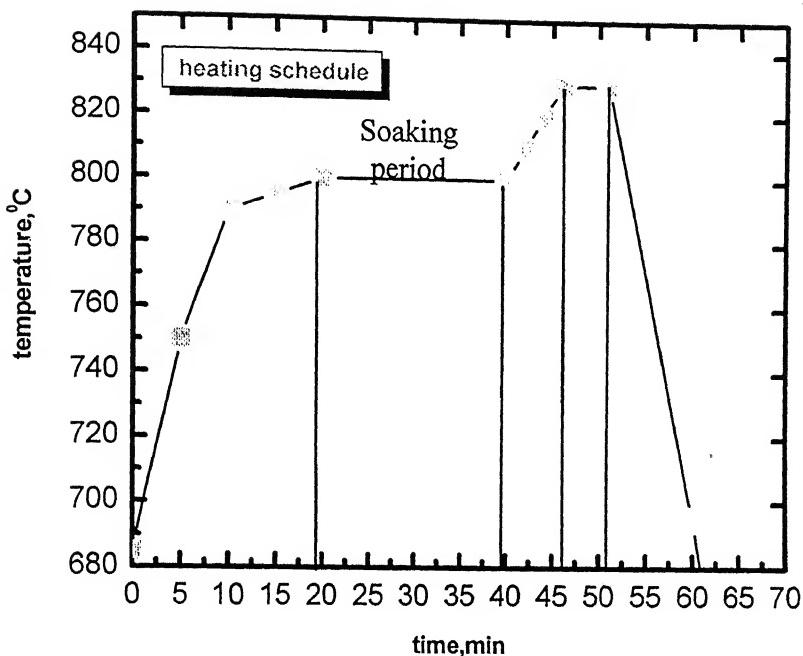


Figure 3.4: The heating schedule followed during the actual brazing process

vii) **Cooling:** The sample was cooled to the room temperature inside the furnace. The hydrogen gas flow was continued to prevent oxidation of the samples. When the temperature reached around 50°C-70°C the furnace door was opened and the samples were allowed to cool in air. The samples were then taken out of the assembly and taken for further testing.

6.2.2 Testing of the samples

i) **Three pairs of cubic samples:** The names given to these three samples were CB1 850, CB2 850 and CB1 830 respectively. The brazing temperature for the first two samples was 850°C while that of the last one was 830°C. The soaking temperature of each of the sample was 800°C and the soaking time was 20 minutes. Each sample was kept at the brazing temperature for 5 minutes. The brazing of CB1 850 was deliberately done by omitting one cleaning step i.e. with HNO₃ acid and without using emery paper roughing

of the samples. In other cases roughing was done using emery papers of size 4. Moreover, all cleaning steps were also followed. The filler metal was used in the form of a foil after cleaning it with soap solution. Prior to placing it in the joint, it was thinned by hammering. The sample shape is shown in Fig 3.5. After brazing of these samples they were analyzed under an optical microscope to study the flow behavior of the filler metal. For analyzing the interface of the joint the samples were cut using a hack saw. Care was taken to prevent excess heating of the sample during the cutting process. An approximate estimation of overflow of filler metal was also done by human eye.



Figure 3.5: The schematic of the cubic sample

ii) Tensile sample fabrication: The names given to the four tensile samples that were prepared are TT1B 850, TT1S 840, TT2S 830 and TT3S 830. The first two samples were prepared initially without success. The design of the assembly is shown in Fig 3.6.1 and Fig 3.6.2. These samples were prepared by brazing two cylinders of diameter 12mm each and length of 45mm. These small cylinders were prepared in a lathe. Sample TT1B 850 was a butt brazed joint using filler metal of the shape of a foil. The foil was pressed between the two cylinders with a jig as shown in the figure. The clearance was maintained by placing a ring of the same material (OFHC copper) between the two cylinders. The clearance was equal to the thickness of the ring which was 50-70 μm . The soaking was done at 780°C for 20 minutes. The maximum bonding temperature was 850°C for 5 minutes. After brazing the sample was cut out in the shape of a tensile specimen having a gauge length of 25mm and diameter of 6mm. Care was taken during machining so that the depth of cut in a lathe was less than 0.1mm else there was a chance of the sample breaking from the joint itself due to intense shear force that was generated during machining in a lathe. All other samples that were brazed were having a step joint instead of butt joint. The braze filler metal used in case of TTS1 840 was also in the form of foil and the same jig of

mild steel was used as in the earlier case. The bonding temperature here was 840°C while soaking was done at 775°C for 20 minutes.

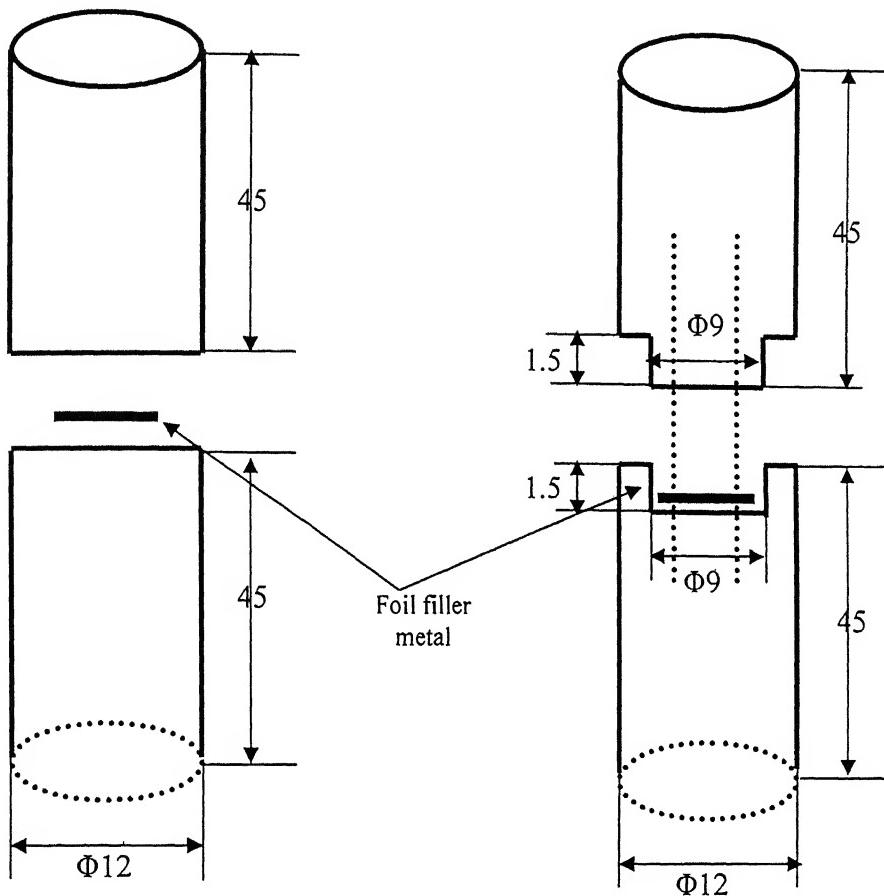


Figure 3.6.1 & 3.6.2: The design of tensile samples TT1B 850 and TT1S 840 with foil as filler metal. Note that no design tolerances were applied.

The other two samples were also step brazed but these used wire filler metal instead of foils. The design of the joint is shown in Fig 3.7. There is some design difference compared to the last ones. Some tolerances were introduced on the surface which forms the path of the filler metal. Since wire filler metal was used for brazing, a gap of 1mm was left between the top and the bottom cylinder. In each case the soaking temperature was 800°C for 20 minutes and the maximum bonding temperature was 830°C for 5 minutes. These samples after brazing were machined in a lathe to bring it into the shape of a tensile sample having total length of 84mm, gauge diameter of 6mm and gauge length of 25mm.

These tensile samples were then tested for their ultimate tensile strength and the place from where the sample fractured was identified.

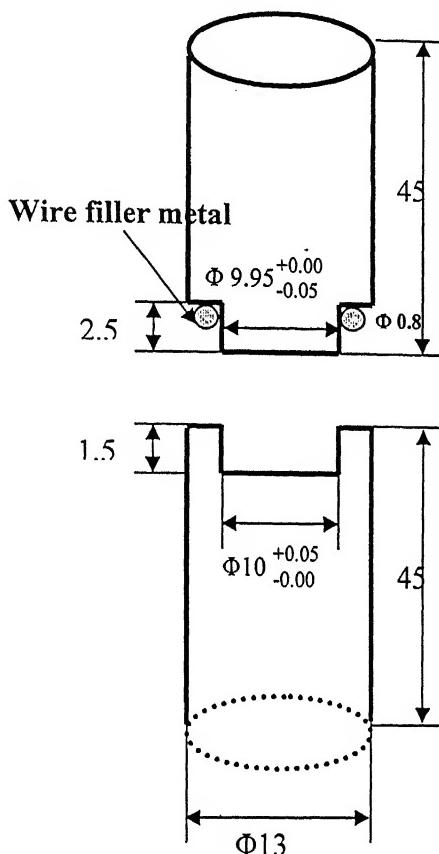


Figure 3.7: The design of tensile samples TT2S 830 and TT3S 830 with wire as filler metal. Note that design tolerances were applied

iii) **Small cylindrical sample for SEM study:** This sample was prepared just in the same way as the tensile samples (step joint), the only difference being the size of this sample was smaller. The total height of the sample after brazing was 2.5cm. The aim of this kind of preparation was to compare the results of the strength of the tensile samples with the flow of filler metal through the joint and its bonding characteristics. Since the method of brazing was similar in both the cases so it was prudent enough to correlate these results. The study of the flow characteristics was done by cutting a section through the interface.

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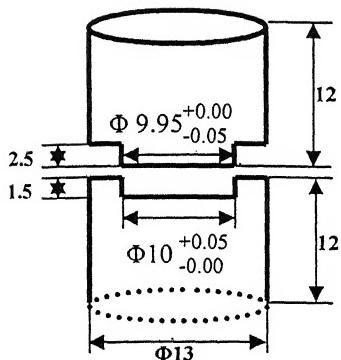


Figure 3.8: Small prototype of the tensile sample which was brazed to study the flow of filler metal by Scanning electron microscopy

iv) Sample preparation to test the vacuum compatibility: The tensile samples were prepared using a particular technique as explained earlier. The strengths of the first two samples were very low as we shall see in the ‘Results and Discussion’ section which would follow after this section. But the strengths of the other two samples were very good. So, SEM study of the previous samples was done to understand the flow of alloy through the joint. But as we know one of the main requirements of the RFQ structure is its vacuum compatibility. This applies to the joints as well. So whatever samples were prepared must have vacuum compatibility. In other words the joints should be leak tight. Hence after the successful brazing of the last two tensile samples it was important to study its vacuum compatibility. But since the tensile samples fractured during tensile testing so a similar sample was prepared in the same way as that of the tensile samples. The only difference in this sample was that it had an attachment at its base to be successfully tested for the vacuum compatibility. The sample is shown in Fig 3.8. After its brazing, which was similar to that of the tensile samples, it was first tested in rough vacuum using acetone and then after 1 hour it was again tested for high vacuum compatibility in Mass leak spectrometer detector (MSLD) using Helium gas. One hour gap was essential since the acetone that was used in rough vacuum testing could have an adverse effect on the results of MSLD testing by blocking the voids in the joints. So if an hour gap is provided, the acetone that could have obstructed the voids will certainly evaporate.

v) Shear test samples: These samples were prepared to study the effect of overlap distance to the thickness ratio required for the failure to occur from the joint. Two plates of 25mm x 50mm x 3mm were machined in a milling machine. Two such plates were then lap joined at specific ratio of overlap distance to thickness of the plates. This joining was

done by brazing at a brazing temperature of 830°C for 5 minutes. Here the soaking temperature was increased to 805°C for 20 minutes. Four such samples were prepared having overlap distances of 2T, 4T, 6T and 8T where T is the thickness of the plates. In each case a load of 100gms was kept over the plates during brazing as shown in Fig 3.9 to keep the plates one over the other. Brazing was done by keeping a foil in between the joints.

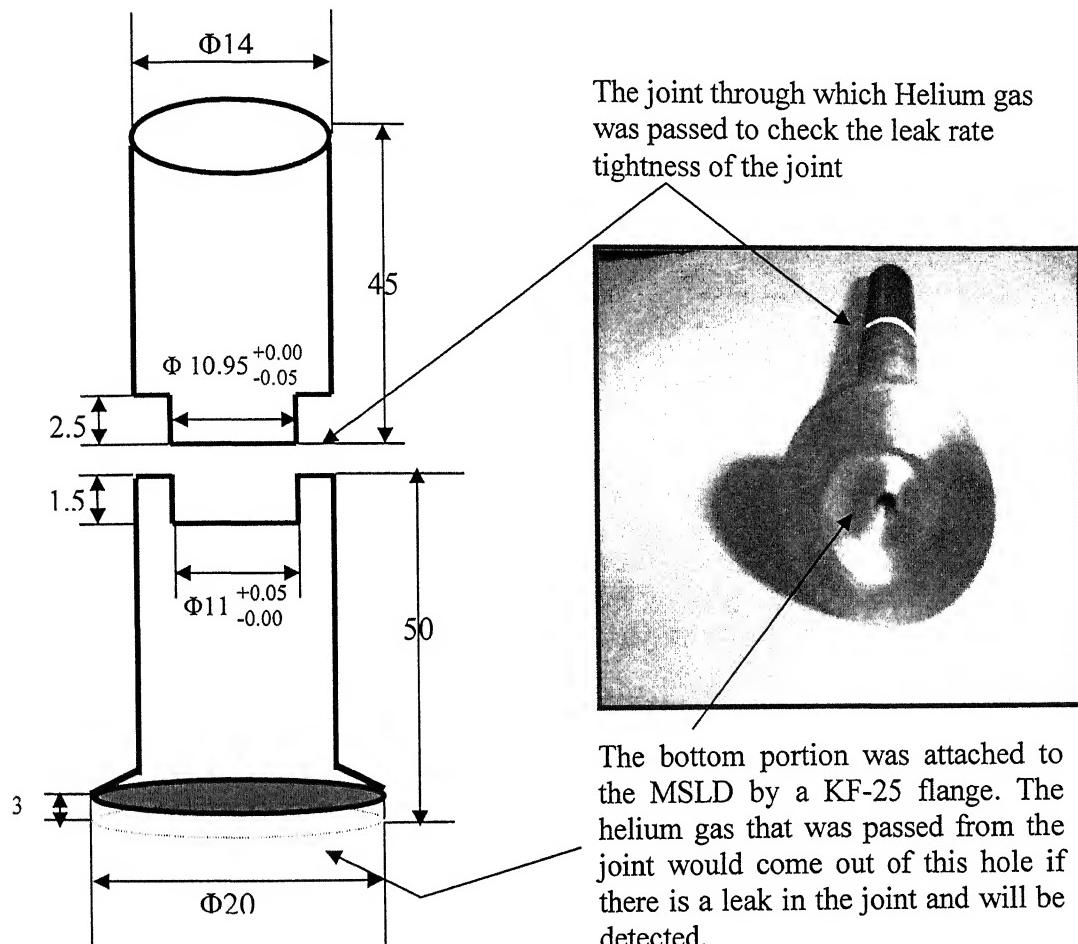


Figure 3.9: The sample that was used for testing the leak rate tightness of the joints in the tensile sample.

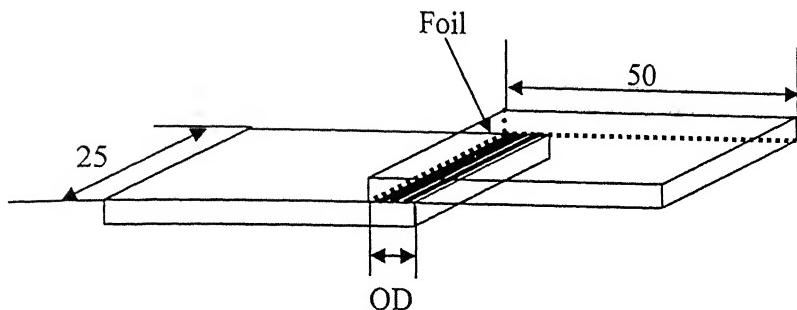


Figure 3.10: The shear testing sample. Here OD means overlap distance

3.1.2 Evaluation processes of the brazed samples

After brazing the samples the following tests were done on the samples to study the quality of the joint.

- Tensile strength evaluation of samples was done in an INSTRON 1195 machine, on tensile samples having a gauge length of 25mm and gauge diameter of 6mm. Shear strength evaluation was also done in the same machine.
- Micro hardness testing was done on a 160mhp microhardness testing machine with a load of 20gm (0.1962N).
- Metallographic studies were done using an image analyzer at various magnifications.
- The flow of the filler material at the joint interface was studied in a JEOL JSM 840A Scanning electron Microscope operating at 15KV accelerating voltage, using secondary electron signal.
- The leak rate testing of brazed joints was done in ASM 122D by passing helium gas through the joint into the vacuum port.

CHAPTER 4**RESULTS AND DISCUSSION****4.1. MECHANICAL PROPERTY DETERIORATION**

The main aim of this thesis work was to study the effect of brazing on the mechanical properties of OFHC copper. Another important aspect that was studied was to develop a brazing methodology of OFHC copper. The brazing filler metal used in the development of the methodology was copper-silver eutectic. But in the actual brazing process of the RFQ structure the filler metal chosen may be different. Hence the study of deterioration of mechanical properties was done over a range of brazing temperature.

It was already mentioned that tensile strength measurements were done at two places: Centre of Advanced Technology (CAT), Indore and Indian Institute of Technology (IIT), Kanpur. The tensile strength along with yield strength measurements were carried out on a MTS machine at a cross head speed of 0.5mm/min. The strength values are shown in the table as follows.

Table 4.1: Tensile strength evaluation done in CAT Indore

Temp, °C	Yield Strength		UTS		% elongation
	kN	MPa	kN	MPa	
UHT(t)*	13.52	87.79	31.79	206.43	60
950(t)*	9.00	52.00	27.93	181.44	50
UHT(L)**	13.59	90.00	33.72	219.05	57.07
800(L)**	16.26	68.00	32.85	213.40	58.44
860(L)**	11.42	66.00	33.90	220.22	59
950(L)**	8.00	58.00	29.76	207.91	51.52

* Transverse section, ** Longitudinal section

Table 4.2: Tensile strength evaluation done in IIT Kanpur

Temp, °C	Yield Strength		UTS		% elongation
	kN	MPa	kN	MPa	
UHT(L)	8.82	71.52	29.43	228.7	60.15
830(L)	12.36	78.0	28.15	228.32	56.45
860(L)	8.87	70.0	28.54	225	62.91
950(L)	8.90	69.75	27.56	216	62
1000(L)	5.52	65.0	18.13	213.45	56.47

Now it is amply clear that during brazing there is a constant decrease of the tensile as well as yield strength of the material with increasing temperature. It is basically due to the continuous recrystallization and grain growth that takes place at high temperatures. We know from Hall-Petch relationship that as the grain sizes increase the yield strength keeps decreasing. But the decrease in the strength of the material is drastic above 900°C, which is about 5%-7%. Therefore it was decided that the use of filler metal having melting range within 850°C is advisable. A buffer of 50°C is necessary since depending on the mass to be heated the soaking time has to be increased. Thus the choice of filler metal of copper-silver eutectic for brazing was made. The maximum bonding temperature was kept at 850°C in all the brazing experiments. When the results from both the places were compared it is seen that the values at the same temperature are quite different. This can be attributed to the fact that the furnace used in the two sets of experiments was different. The experiments done in CAT Indore were done in bigger furnace having a heating rate of 700°C/hr while that used in IIT Kanpur had a heating rate of 150°C/hr. Moreover the difference can also be attributed to some extent to the differences in the dimensions of the samples.

This same trend was also noticed when the hardness values were evaluated from the smaller samples that were also kept along with the tensile samples. Now the two sets of hardness values would be tabulated as follows.

Table 4.3: Hardness variation with temperature IIT Kanpur

Temperature	VHN
UHT(L)	82.17-97.9
830(L)	70.0-84.0
860(L)	69.78-83.5
950(L)	67.28-82.4
1000(L)	66.0-82.17

The variation of the mechanical properties is now being shown in a graphical manner.

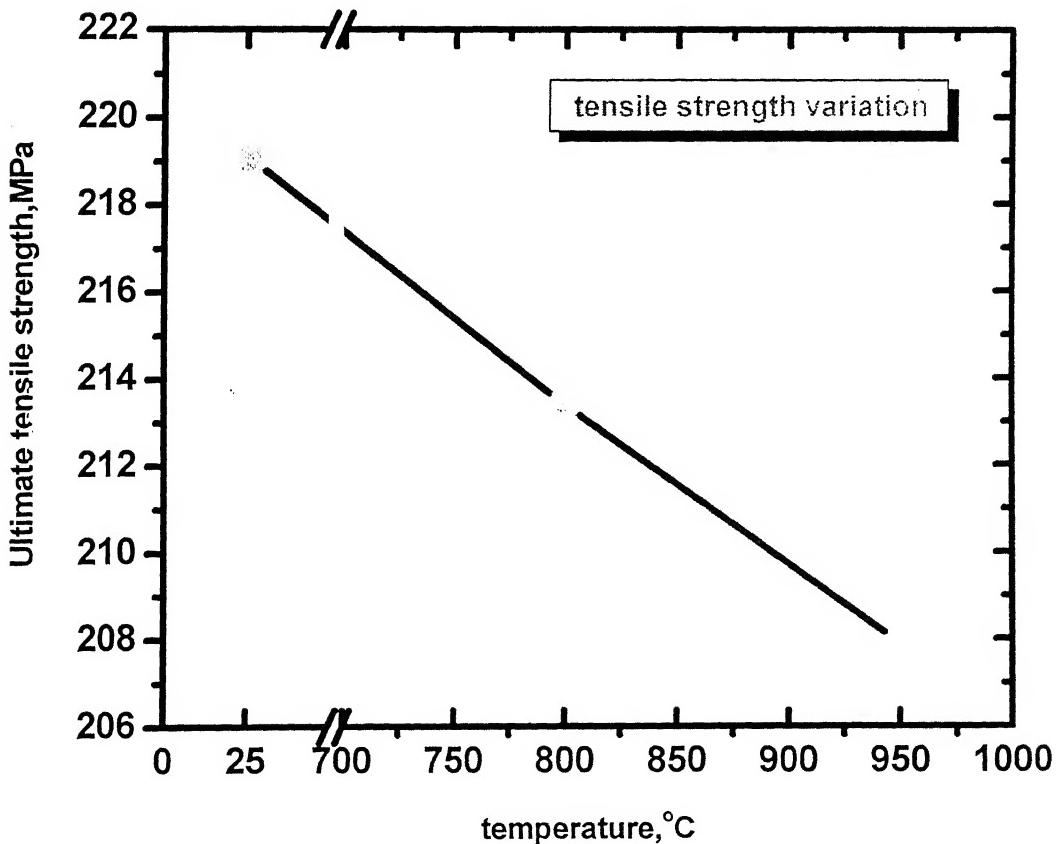


Figure 4.1: The tensile strength variation with temperature is shown for the first set of experiments done at CAT Indore

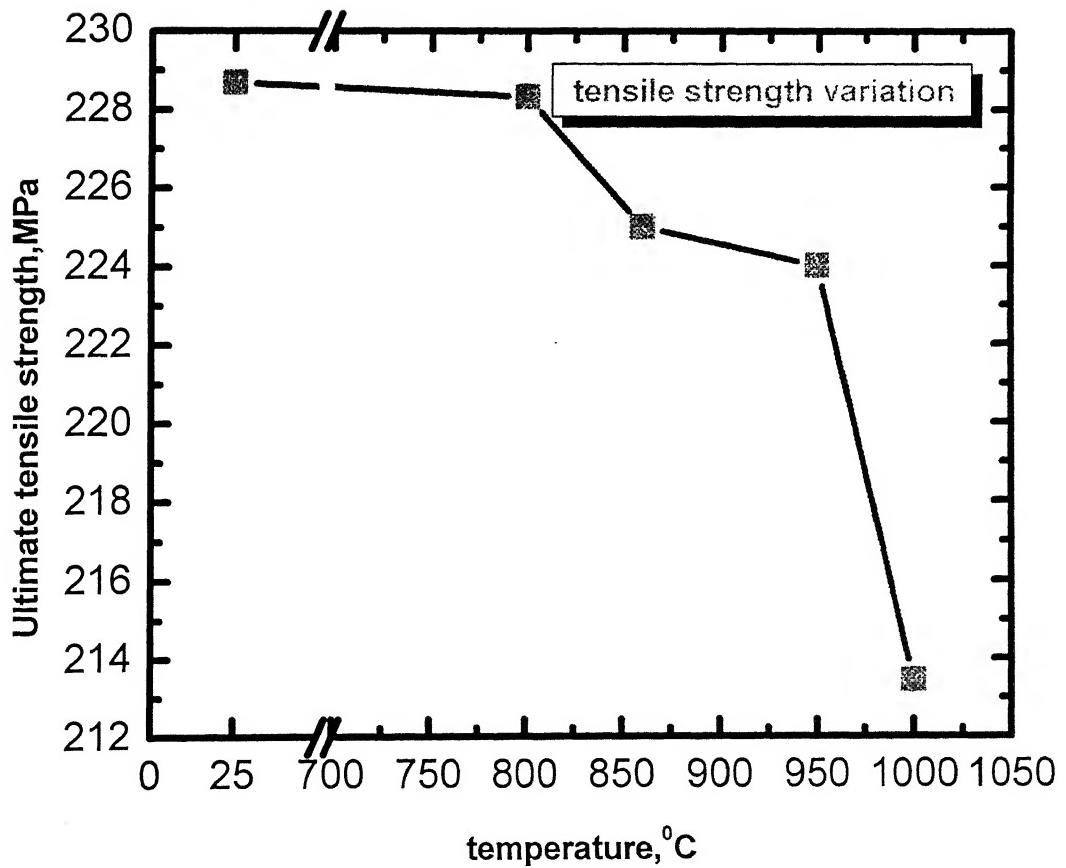
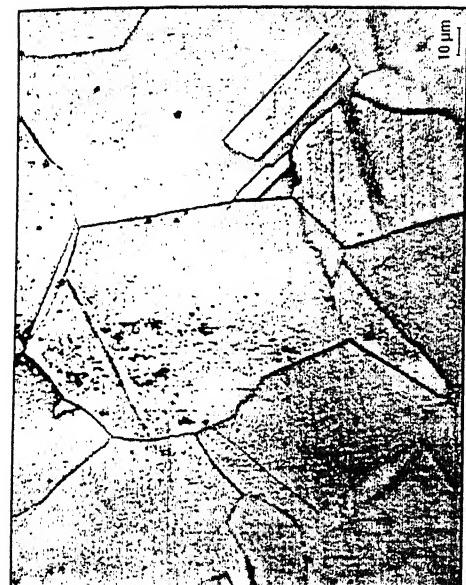
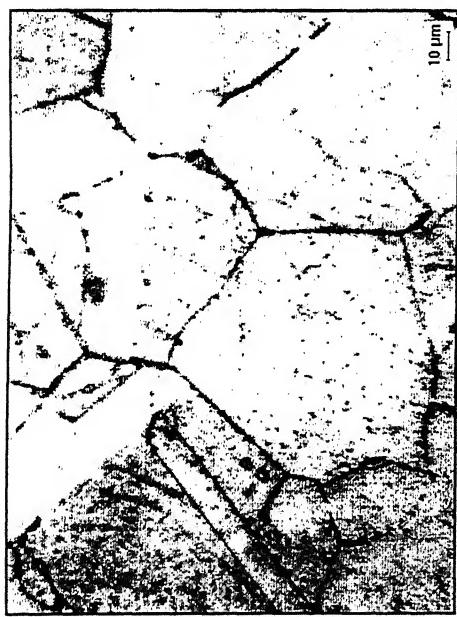


Figure 4.2: The tensile strength variation with temperature is shown for the first set of experiments done at IIT Kanpur



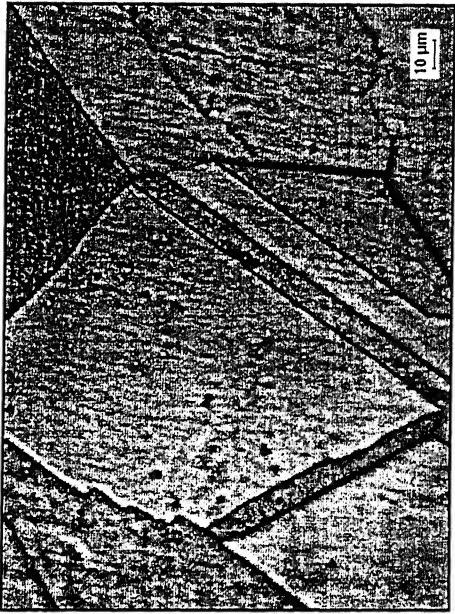
At 25⁰C



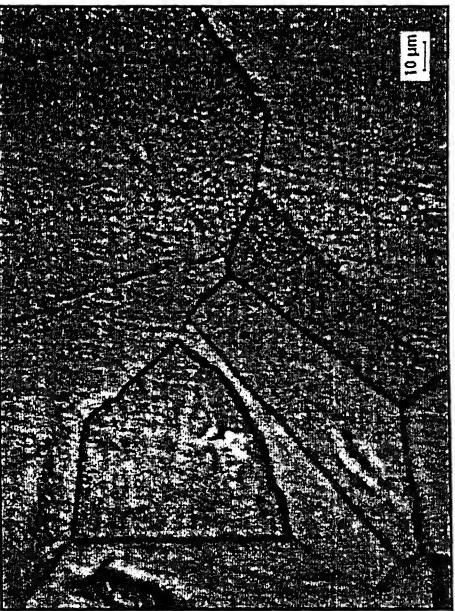
At 800⁰C



At 860⁰C



At 950⁰C



At 1000⁰C

Figure 4.4: The figure shows grain growth with temperature at a magnification of 500X

4.2 RESULTS OBTAINED ON THE BRAZED SAMPLES

From the study of mechanical property deterioration, as a consequence of temperature of brazing, we now move on to analyze the results of actual brazing experiments. In the subsequent pages the experiments done, will be explained in a chronological manner so as to give a better understanding of experiments undertaken. Our initial aim was to study the joint strength of a brazed sample. So two tensile samples, TT1B 850, TT1S 840 were prepared. Tensile strength evaluation was done in an INSTRON 1195 machine. The tensile strength of a planar joint is its ability to sustain stress applied perpendicular to the joint without breaking. The measured strength will be sensitive to the geometry of the test pieces and of the joint, particularly if the latter is comparable in strength to parent material and if the filler has slight elasticity, a point that needs to be kept in mind when comparing strength data [West et al. 1971; Trimmer and Kuhn 1982]. The results of the testing are summarized as follows.

- i) The tensile strength was much lower than the base metal strength. It was 25.2 MPa in case of TT1B 850 and 72.3 MPa in case of TT1S 840.
- ii) The failure occurred from the brazed joint. The SEM micrograph of the fractured surface shows faceted surfaces, shown in Fig 7.5.

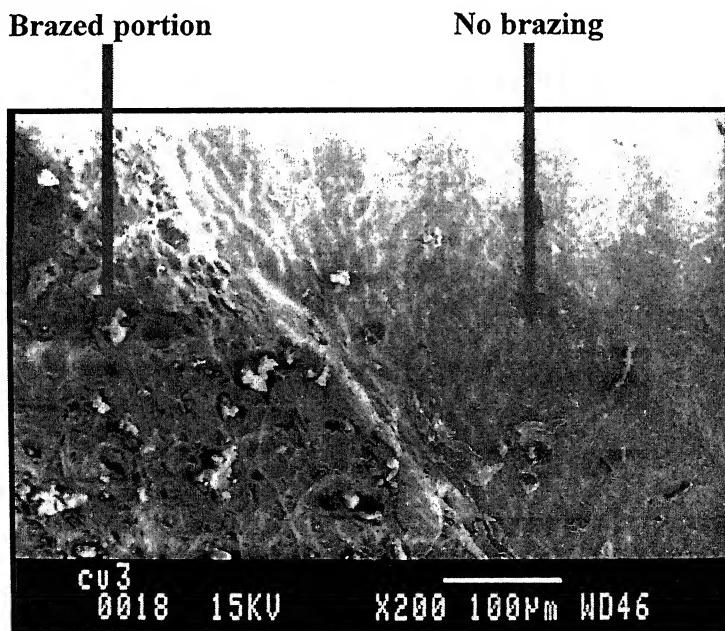


Figure 4.5: Butt joint failure during tension test as seen in SEM. No flow of filler metal inside the joint.

The results indicate that despite all precautions, some designing and process errors have crept in during the actual brazing process. The reasons for improper brazing were later identified to be the following ones.

- i) A clearance of $70\mu\text{m}$ that was provided between the faying surfaces of the joint resulted in no capillary effect. As a consequence, major proportion of molten filler metal flowed out of the joint. The purity of the furnace atmosphere being of a very high order also improved the flowability of the filler, and this can also be a reason for the overflow.
- ii) The pressure that was applied by the fixture to keep the two cylinders in vertical alignment, during the brazing process, was very high. This also helped in squeezing the filler metal out of the joints. In other words the load applied to keep the cylinders in vertical position was too high.
- iii) These cylinders were machined in a lathe. The joint surfaces were thus given facing operation in the lathe to remove unnecessary burrs and protrusions. But during the facing operation the speed of lathe at the centre of the cylinder face becomes zero and as a result the cutting is not possible at the centre. Thus a protrusion remains at the centre which is called the high point. So when these cylinders are supported vertically one over the other, this high point makes the upper cylinder imbalanced. This gives a translatory motion to the upper cylinder in the horizontal direction. This motion is called play of the upper cylinder. Due to this motion the filler metal, when molten, accumulates on one half side of the total joint surface area. This means one side does not braze at all and the result is a poor joint. This is what exactly happened in the sample TTIS 840. This could have also happened in TTIB 850 but it could not be deciphered since in this case the total filler metal overflowed from the joint surface whose photograph is shown in Fig 4.6.

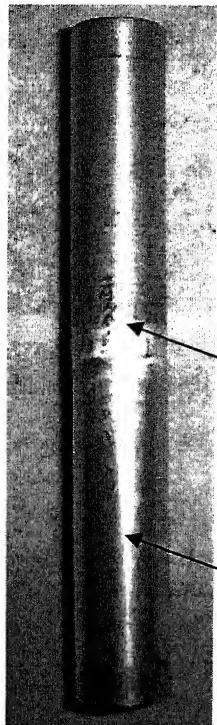


Figure 4.6: The brazed TT1B 850C is shown in figure where the total filler metal has overflowed from the joint

The position where the two cylinders
were joined

Overflowed filler metal

- iv) Finally the brazing temperature in both cases was a bit too high which increased the fluidity of the filler to such an extent that the filler metal overflowed out of the joint.

The failure to braze the tensile samples made us rethink our approach. Thus it was decided that the brazing should be done on small samples first which would provide the necessary data for brazing bigger samples. To analyze whether brazing of the smaller samples has been proper or not we have to develop ways to identify it. It has been mentioned in most of the literature, on brazing, that the analysis of the joint interface through optical or scanning electron microscopy serves a good standard for evaluating the joint quality. In this method what we do is to cut a section through the joint and then polish the surface in which the brazing joint can be visualized. After polishing the sample it is etched slightly and is seen under an optical or scanning electron microscope. The interface between the base metal and the filler in the joint is observed under the microscope. If the filler metal is visible consistently

from the starting to the end of the joint then we conclude that the flow of the filler metal has been proper. This in turn means that the joint quality is good. But such a study needs to be done in at least two sections of the same sample to corroborate the fact that the joint quality is good. As it was mentioned earlier three small cubic samples were brazed to develop the idea of the procedure to be followed for brazing. These samples were coded CB1 850, CB2 850 and CB1 830. In each sample some definite study was conducted. Two main studies conducted were the effect of removing one cleaning step and the effect of high temperature. Since the brazing involved no jigs, any external pressure was also missing on the samples.

In case of the first sample the cleaning step with acid was deliberately done away with to study its effect. The optical micrographs of the sample are shown in figures 4.7 to 4.11.



Figure 4.7: The optical micrograph shows good flow of filler metal at a magnification of 200X. The photograph was taken after etching it with aqueous ferric chloride

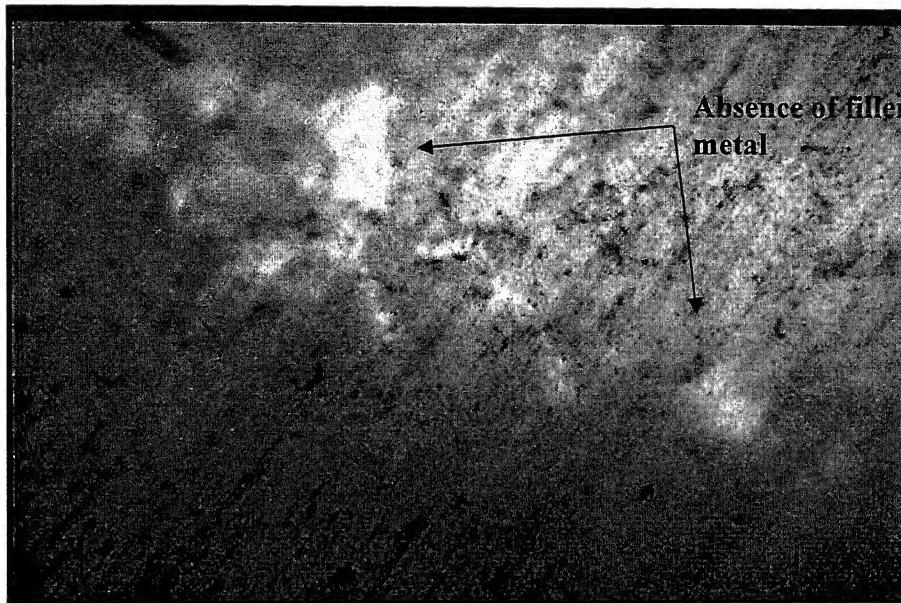


Figure 4.8: The optical micrograph shows no flow of filler metal at all through the joint. The photograph was taken without etching

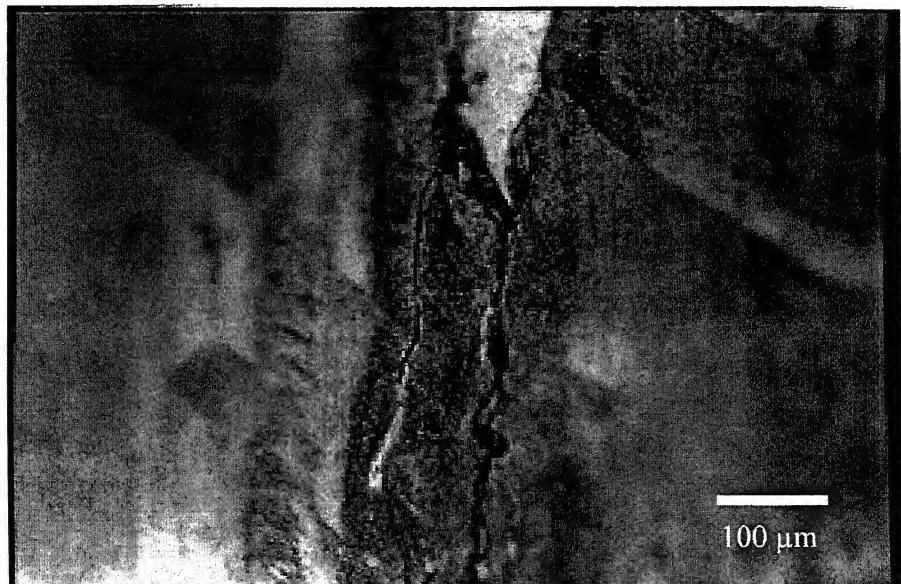


Figure 4.9: The optical micrograph shows no flow of filler metal at all through the joint. The photograph was taken after etching

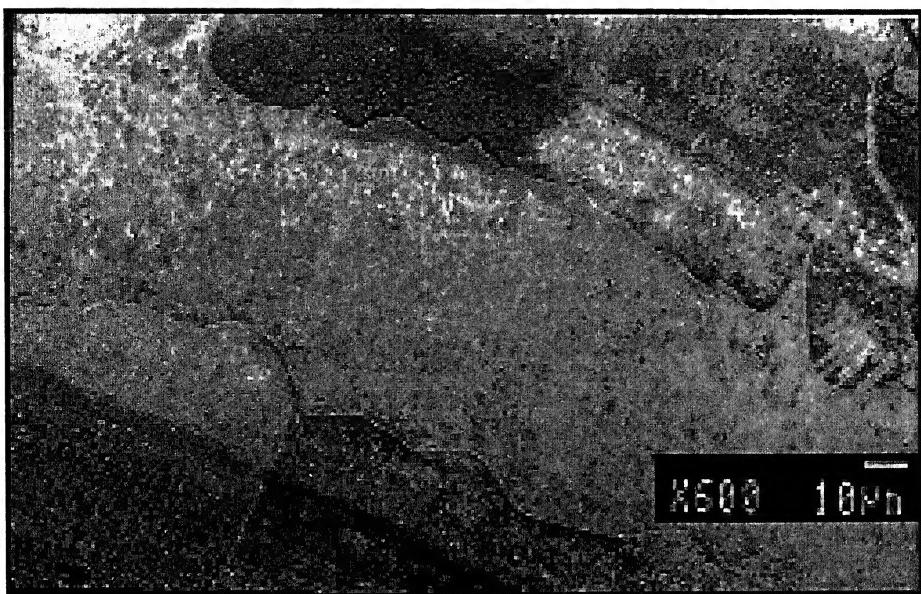


Figure 4.10: The Scanning Electron micrograph shows good flow of filler metal through the joint. The photograph was taken after etching

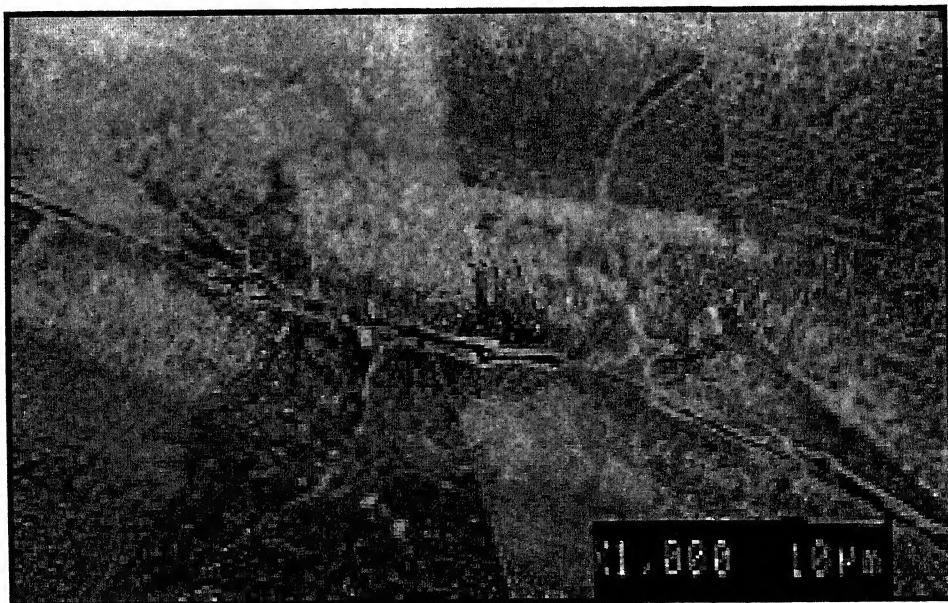


Figure 4.11: The Scanning Electron micrograph shows no flow of filler metal (even at 1000X) at all through the joint. The photograph was taken after etching.

The results of brazing can be inferred from the micrographs that have been presented. Two sets of micrographs are presented; one from the optical micrographs and the other from the

scanning electron micrographs. The photographs from the SEM give us a better idea about the brazing process, as we can use much higher magnifications to analyze the existence of flow of the filler metal. The following points can be inferred from it.

- i) The brazing has been good at some places while at some sections there has been no brazing at all. Thus it means that there was some oxides and dust still sticking on the surface which disallowed the filler metal to flow through those regions. Hence no brazing was seen at some sections while at others it was visible. Thus cleaning was seen as the most essential step in the whole brazing process.
- ii) After the sample CB1 850 was taken out of the furnace it was observed that there has been slight overflow of the filler metal from the joint on the side faces of the workpiece. This can definitely be accounted for as due to the high temperature of brazing. This needs to be examined again in the next sample CB2 850.

The second sample was now tested in a manner similar to the earlier one. The only difference in its joining process was that it was cleaned following all the steps. The photomicrographs are shown in Figures 4.12 and 4.13.

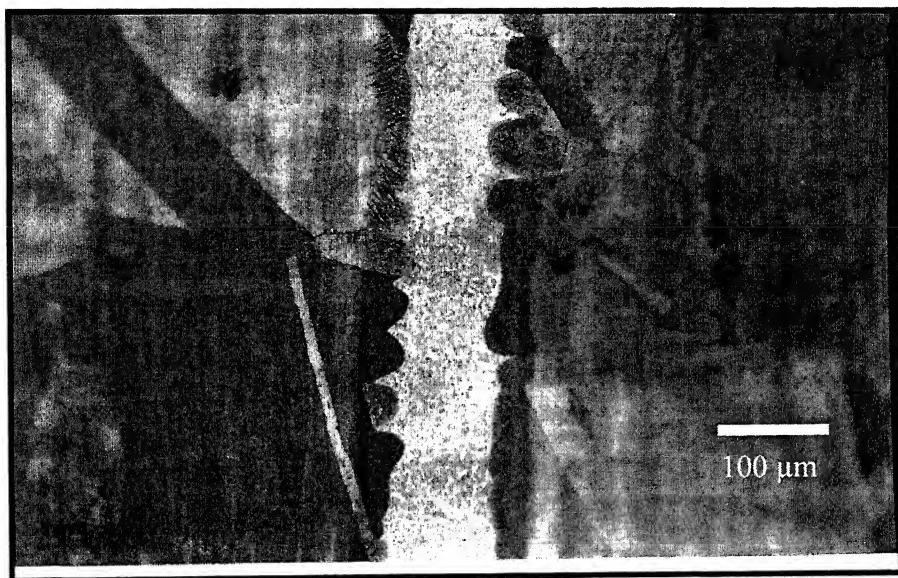


Figure 4.12: The optical micrograph shows good flow of filler metal at a magnification of 200X. The photograph was taken after etching it with aqueous ferric chloride

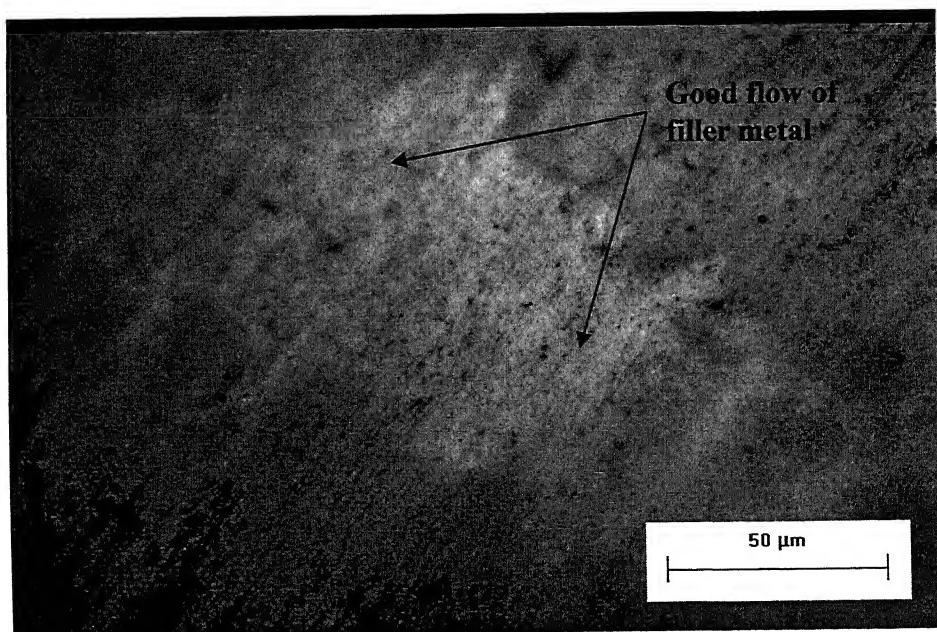


Figure 4.13: The optical micrograph shows good flow of filler metal at a magnification of 500X. The photograph was taken without etching

The only difference that was noticed in this brazed sample was that the flow of the filler metal throughout the joint has been uniform. Hence it can be once again emphasized that cleaning of the sample is a very important step. As usual, the overflow of filler metal still exists. This means we can now confirm that the overflow is due to the high brazing temperature. Hence we concluded that when we use BAg-8 as filler metal the maximum brazing temperature has to be below 850°C. Therefore, we now brazed our last sample CB1 830 following identical processes as the earlier samples. The only change was in the brazing temperature that was brought down from 850°C to 830°C. The photomicrographs of the last sample showed very good results as would be confirmed by the figures 4.14 and 4.15.

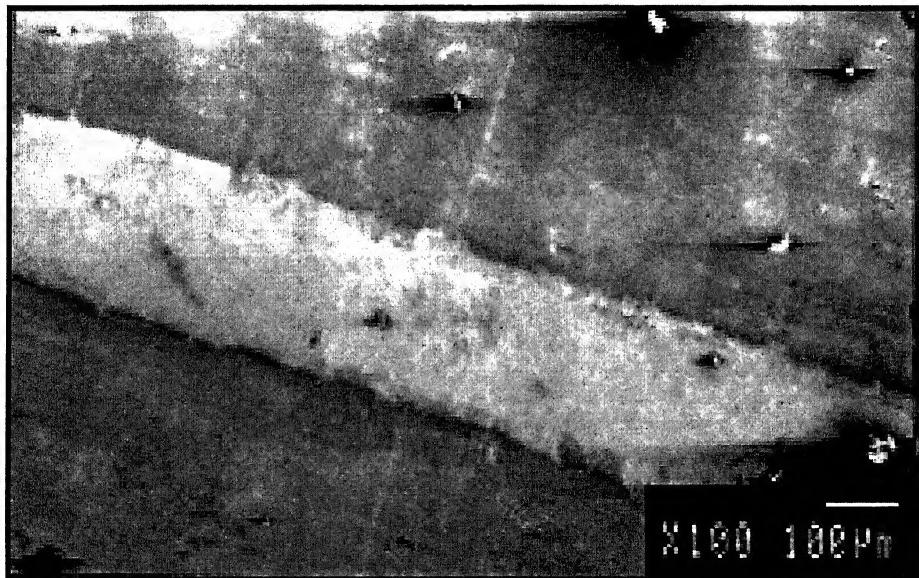


Figure 4.14: The SEM photo shows uniform flow of filler metal through the joint. The sample was slightly etched

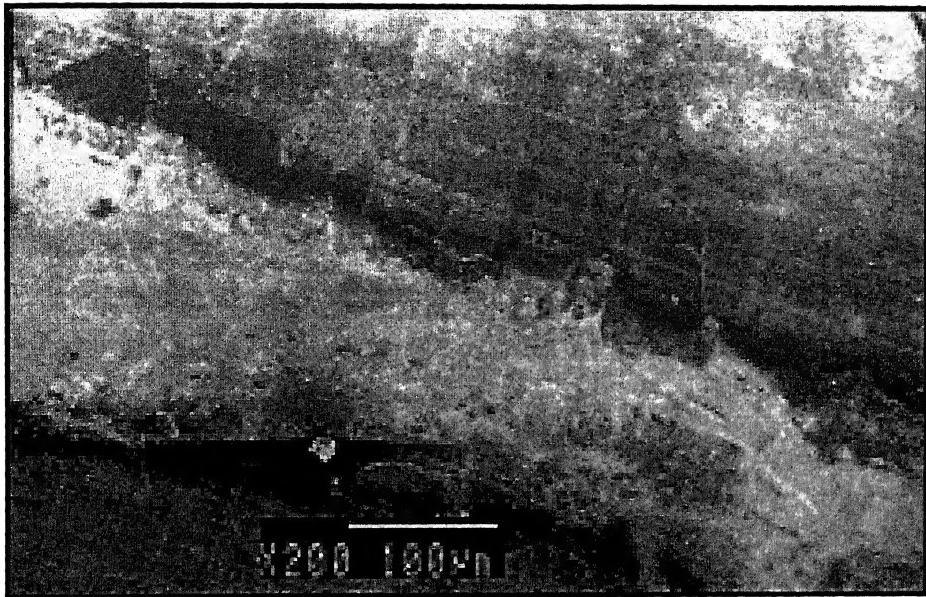


Figure 4.15: The SEM photo shows almost uniform flow of filler metal at another location of the sample. The sample was slightly etched

These results provided us the understanding of the processes involved in brazing of OFHC copper with BAg-8 as the filler metal. The following points need to be considered.

- i) The soaking temperature should be 800°C for at least 20 minutes.
- ii) The brazing temperature should not be greater than 830°C, and brazing time should be less than 5 minutes.
- iii) The cleaning steps should be followed carefully.
- iv) Excessive pressure should be avoided on the joints.
- v) Clearance between the joints should be almost zero. In other words a close fit up design should be preferred.
- vi) External fixturing should be limited to bare minimum.
- vii) Designing of the joints should be considered with seriousness.

After developing the proper methodology we proceeded with our earlier task of processing and evaluating the tensile strength of the joint in a brazed sample. This was considered very useful as this will also give us further idea of brazing the prototype sample of RFQ structure having good joint quality. The design of the sample was already shown in our previous section (please refer to Fig 3.7). The filler metal used in this case was the wire type

which was wound around the circumference as shown by the arrows in that figure. The idea was that as the temperature increase the gas inside the joint would expand and try to come out through the gaps leaving almost no gas inside the joint. In the meanwhile the filler metal remains solid till soaking period. Now as the temperature is further increased the filler metal melts and tries to enter the joint due to the capillary action. After the temperature reaches its maximum the cooling is started after 5 minutes. At this moment the gas starts contracting thereby creating a negative pressure inside the joint which helps in sucking the liquid filler metal into the joint. This is what was done and two such samples were prepared. These samples were then carefully machined in to tensile shape in a lathe machine. The samples were then tested in INSTRON 1195 testing machine at a crosshead speed of 0.5mm/min. The results of tensile test are tabulated along with the values of earlier samples as follows.

Table 4.4: Results of tensile testing

Sample	Soaking temperature, °C	Soaking time, min	Brazing temperature, °C	Brazing time, min	Tensile strength, MPa
TT1B 850	800	20	850	5	25.2
TT1S 840	800	20	840	5	72.3
TT2S 830	800	25	830	5	207.2
TT3S 830	805	30	830	5	205.9

**Failure at the joints****Failure in the base metal**

The first two samples had failed from the joints, while the next two failed from the base metals. This fact would now be proved through the SEM photographs of the fractured surfaces.

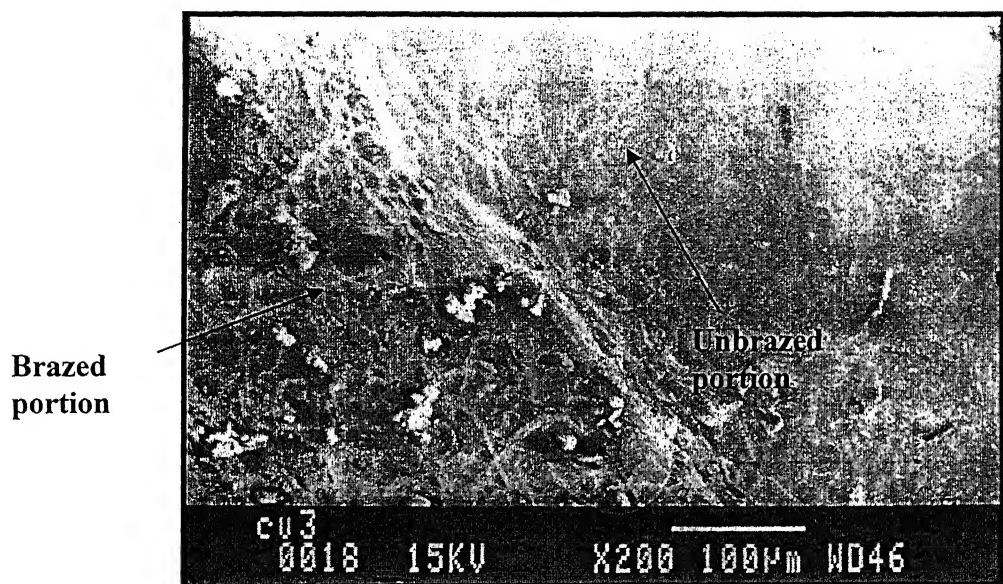


Figure 4.16: SEM photo of the fractured surface of TT1S 840

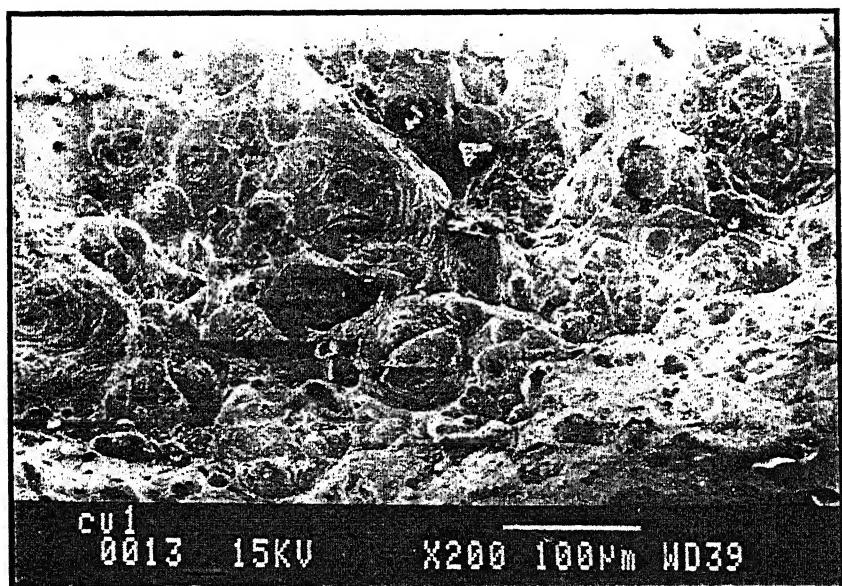


Figure 4.17: SEM photo of the fractured surface of TT2S 830 which failed through ductile mode fracture from the base metal

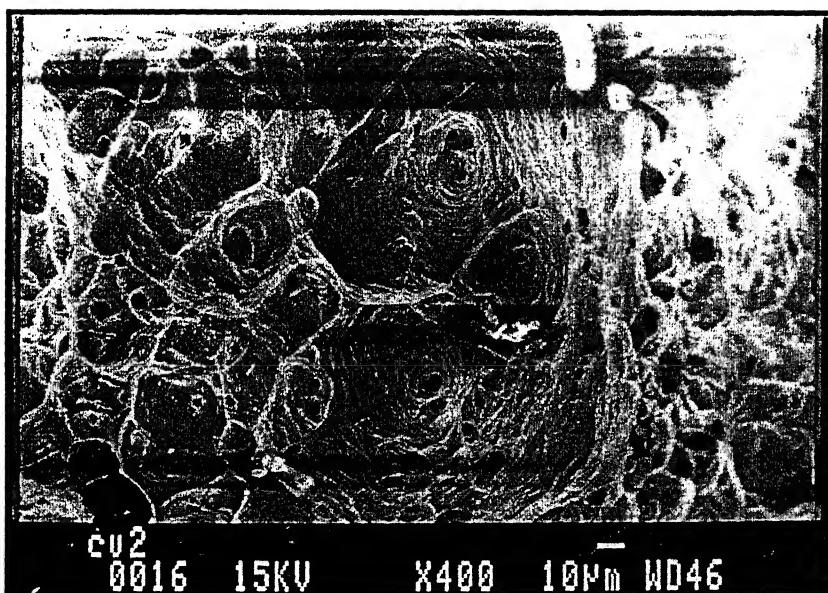


Figure 4.18: SEM photo of the fractured surface of TT2S 830 which failed through ductile mode fracture from the base metal

The joint strength in the latter case improved dramatically after few alterations in the design and the process of brazing. As discussed earlier, the filler metal was drawn into the joint by capillary force as well as by the negative pressure created in the joint by the expansion of gases. As the bonding of the joint was stronger the failure occurred from the base metal. This resulted in the strength of the joint being greater than that of the base metal.

The failure occurring from the base metal does not guarantee that the flow of filler metal into the joint has been uniform. Thus our task is to analyze such joints by the following methods.

- i) Study of the joint cross section by metallography and SEM techniques.
- ii) Checking the condition of leak tightness of joint by Mass spectrometer leak detection technique.

The metallographic study was carried out on a small sample that was brazed in a manner similar to the tensile sample having the same types of joint, using wire form of filler metal wound around the joint. The only difference was that the sample was not as long as the tensile sample. A section was cut across the joint after brazing and SEM analysis was done on

that joint. The micrograph shown below clearly suggests that the flow of the filler has been proper throughout the joint.

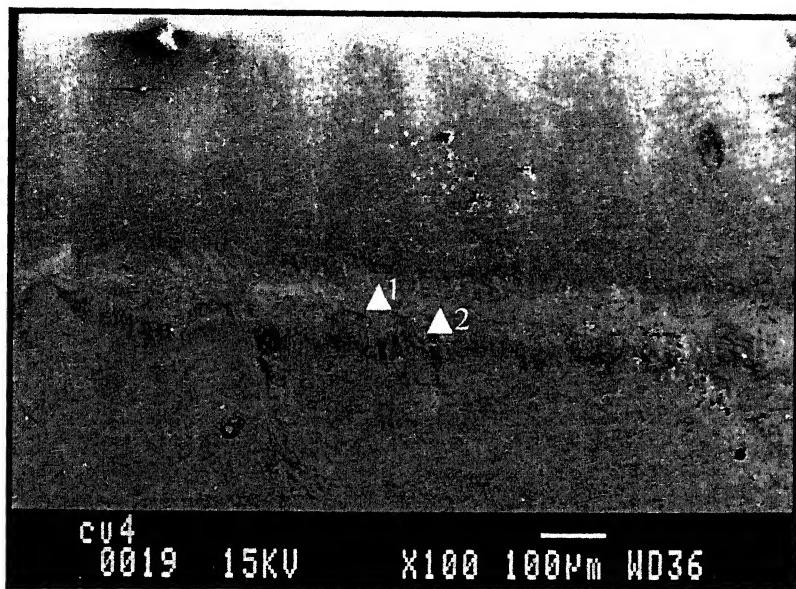


Figure 4.19: SEM photo of the interface of the base metal and the filler metal of a small prototype of the tensile sample

Two more analyses were carried out at the interface namely, hardness profile across the joint and compositional analyses at points 1 and 2. It is a fact that due to the mutual solid solubility of the base metal in the filler composition there is bound to be some inter-diffusion, during brazing, that would also lead to variation of hardness across the joint depending on the depth of diffusion. This can also be concluded by studying the composition at two points; one just near the joint interface and the other one inside the filler metal. These analyses would further confirm whether the bonding has been strong between the filler and the base metal. The hardness profile across the interface and the composition analysis is shown in figures 4.20, 4.21 and 4.22.

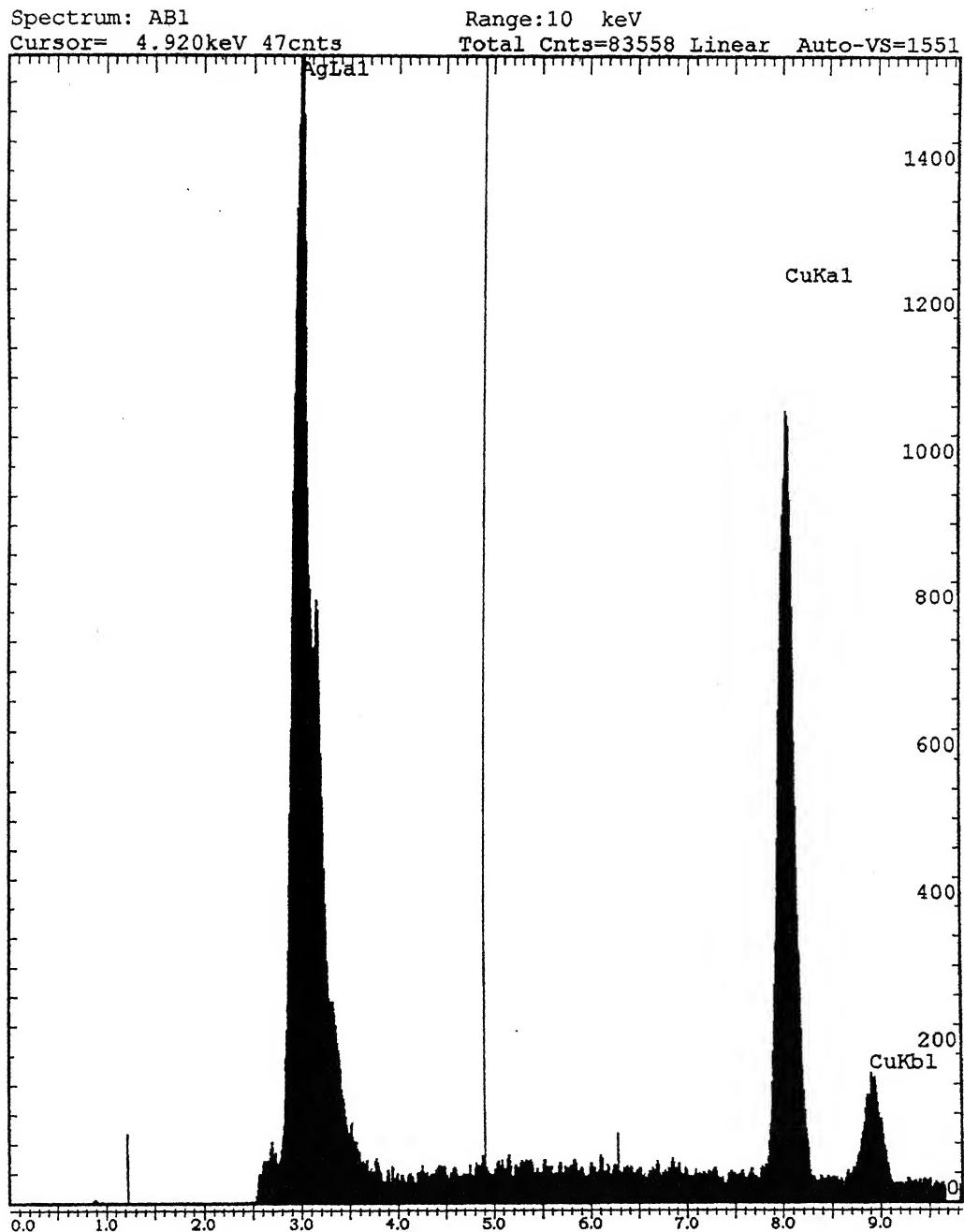


Figure 4.20: The compositional analysis of point 1 gives the actual composition of the filler metal

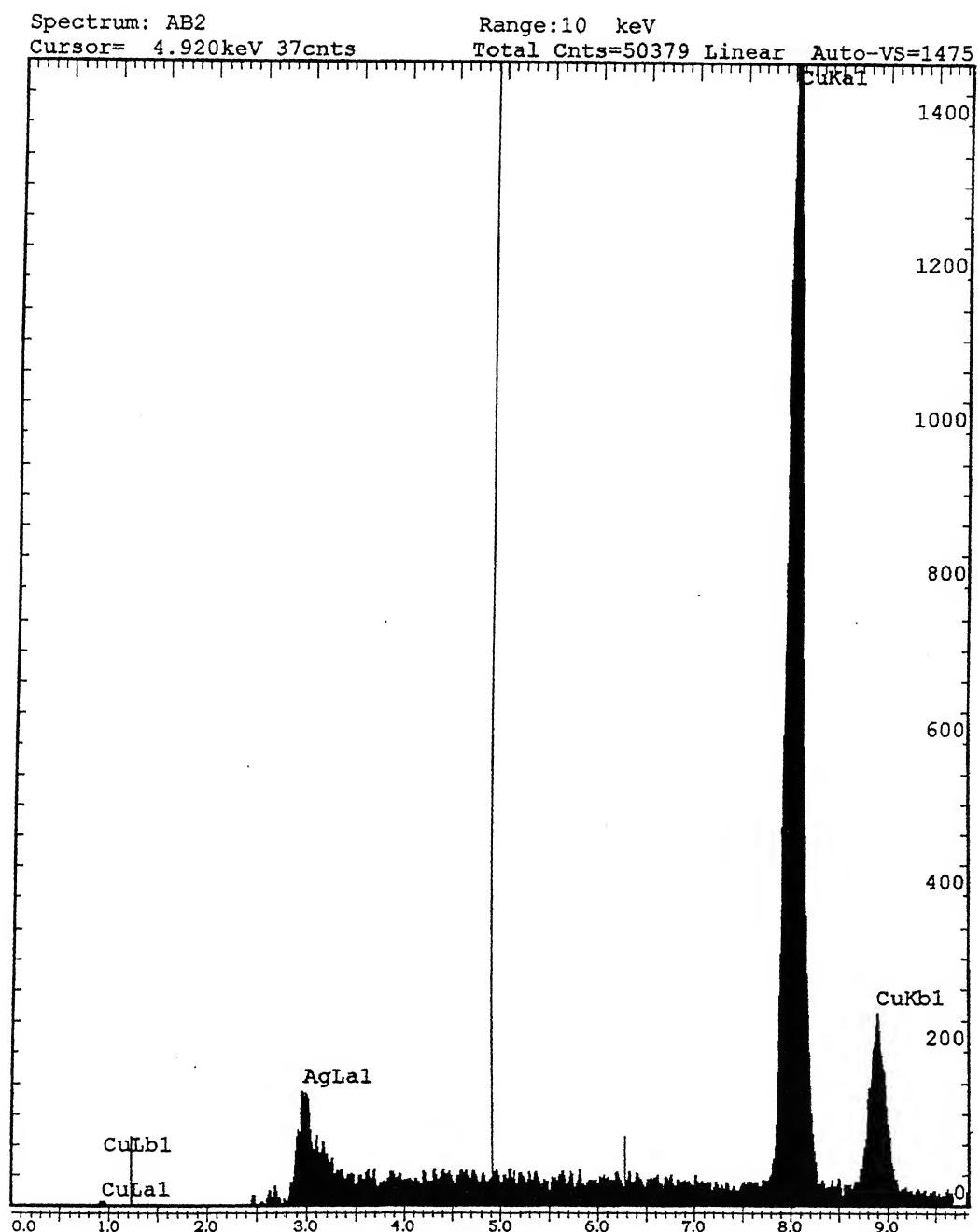


Figure 4.21: The compositional analysis of point 2 which suggests that there has been some diffusion of copper into the filler metal.

The compositional analyses at the two points are given in table 4.5.

Position	Cu, wt%	Ag, wt%
1	72.62	27.38
2	97.56	2.44

Table 4.5: The compositional analysis at points 1 and 2.

Now we look into the hardness profile and compare it with the compositional analysis.

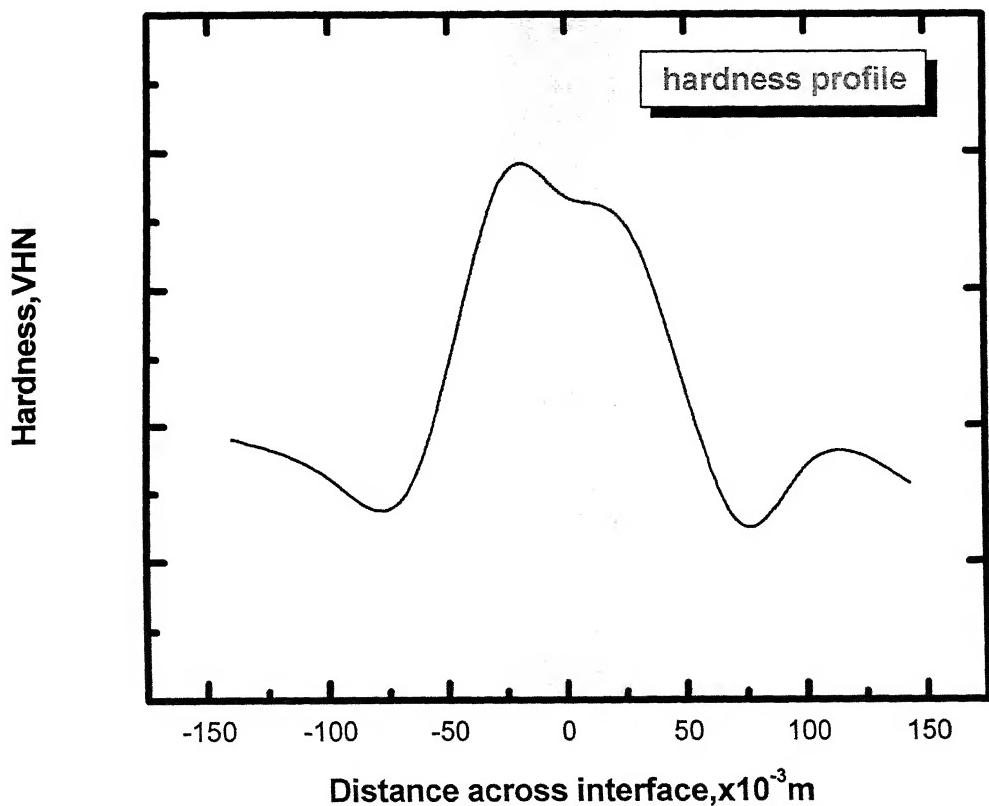


Figure 4.22: The hardness profile across the interface is shown. The shaded portion shows the interface region.

One more sample was prepared for testing the leak tightness of the joint in the tensile sample. As shown in the figure 3.9 the design of the joint and its processing is almost similar to the tensile samples. But a conical base has been machined instead of a cylindrical one as in the tensile samples. This was done so that it can be attached into the RF flange which is used for connecting it to the vacuum port of the MSLD. The brazed sample was then tested for the leak tightness in the MSLD by passing helium gas. It was found that a leak rate higher than 10^{-10} atm-cm³/sec was achieved.

Thus it was concluded that the joint processed by the latest brazing method was satisfactory.

Joints in brazed samples are basically lap joints; these experience large shear force during functioning. Therefore, our last set of experiments was aimed at calculating the minimum overlap distance that is required in a brazed lap joint so that the failure occurs at the base metal and not the joints. This overlap distance was varied by keeping the thickness of the workpiece constant. The samples were prepared according to the standard prescribed in AWS standards. But the testing could not be carried out according to the standards due to nonavailability of the testing attachments in a tensile testing machine. So a slight modification was done in the testing of the shear samples. The samples were prepared as explained in the previous section and the shear strength testing was done by holding the two sides marked A and B as shown in figure in the clamps in tensile testing machine. The sample was then pulled in tension and the shear strength was calculated.

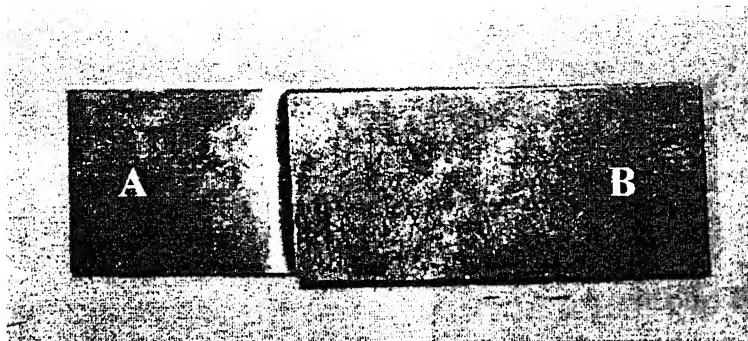


Figure 4.23: A and B are the positions where the samples were held in the clamps of the tensile testing machine

The results of the shear testing are plotted graphically in figure 4.24.

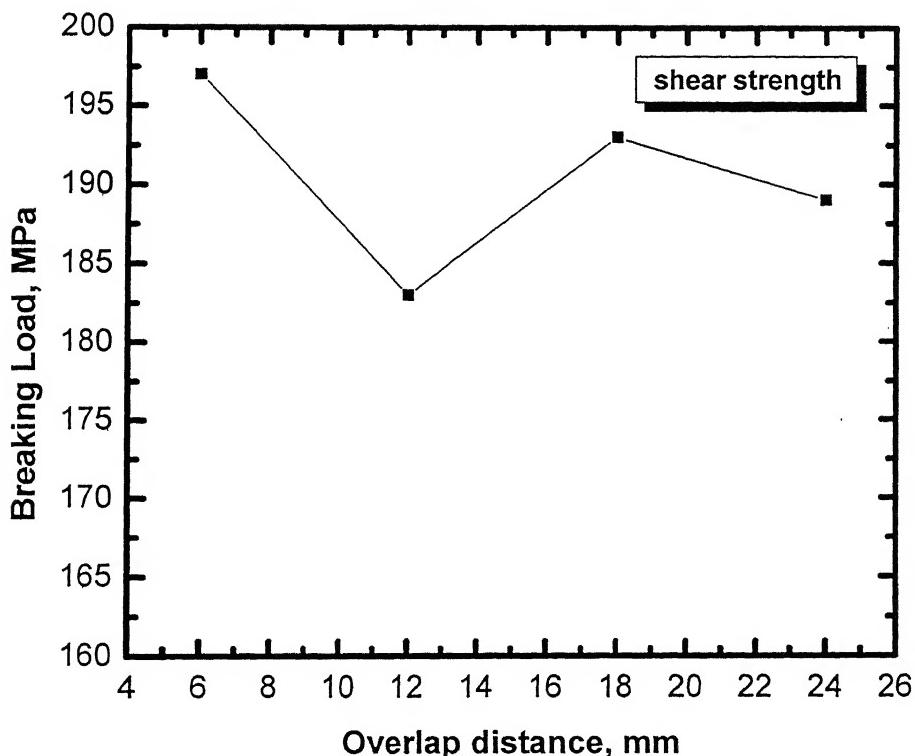


Figure 4.24: Graph showing the minimum load upto which the joint can withstand shear load. In each case the failure occurred at the base metal. Although some tearing was observed at the joints when at minimum overlap distance.

The results indicate that the failure in each case occurred at the base metal through ductile mode. But in the case where the overlap distance was just twice the thickness of the workpiece, some tearing at the joints was also observed during the testing process (please see figure 4.25).

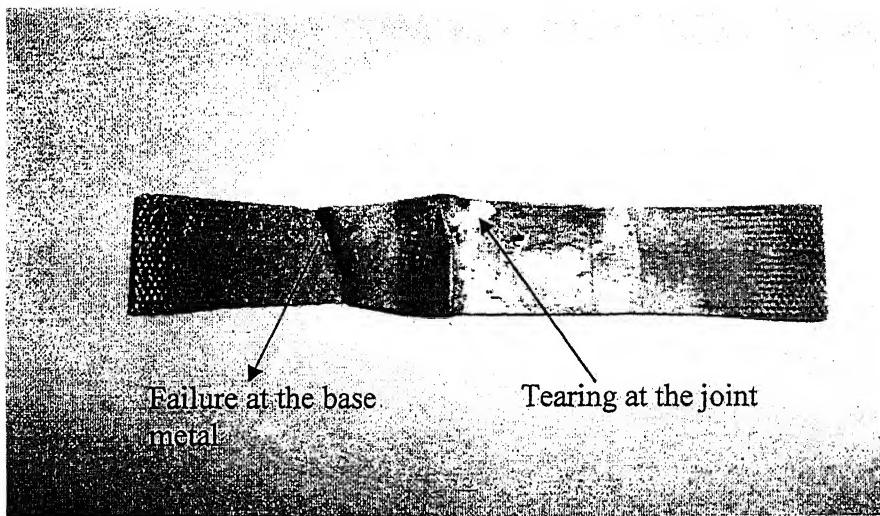


Figure 4.25: The shear sample having overlap distance of $2T$ has failed from the base metal although some tearing at the joints are seen.

CHAPTER 5

CONCLUSIONS

The project consisted of two parts. The first part analyzed the deterioration of the mechanical properties of OFHC copper due to brazing and ways to remedy this problem. The second part was concerned with the characterization of the joints, brazed copper-silver eutectic filler metal.

The reduction in the tensile and yield strength with increasing processing temperature was a natural outcome of high temperature brazing operation. The reason is attributed to the recrystallization and grain growth that occurs whenever any material crosses its recrystallization temperature which is above $0.6T_m$, where T_m is the melting point of the concerned material in kelvin. It is well known that as the grain size increases the strength of the material decreases according to Hall-Petch equation.

The methods to prevent the deterioration of properties can be classified into two groups. One method is to modify the alloy compositions which could prevent grain growth, without sacrificing on the primary properties of OFHC copper. The other method is to modify the process such that the reduction in strength can be minimized.

In this connection, two important alloying additions could be silver and alumina particles. The effects of both these additions have been already discussed in the literature survey section. Fine alumina particles dispersed within OFHC copper would affect the electrical or thermal properties of the material only a little, but would definitely help in retarding the grain growth during annealing. Such materials are being used at few places around the world in place of OFHC copper for this type of application. One such material is known by the trade name GLIDCOP.

The second method of reducing the deterioration in mechanical properties is to reduce the heating rate during the brazing process. It can be confirmed by the experiments that were done during this project work. As already mentioned, two different sets of heating rates for tensile testing samples were used: the first one having a rate of $700^{\circ}\text{C}/\text{hr}$ while the second was around $150^{\circ}\text{C}/\text{hr}$. The results indicate that the reduction in tensile and yield strength occurs as the heating rate is reduced. In one of the papers by Brian et al. at Army Research Lab,

Aberdeen, the effects of high temperature, medium strain rates, and high heating rates on the stress-strain results were mentioned. It also mentions that a decrease in yield stress is observed as the heating rate was increased, although the maximum temperature was limited to 375°C.

The second part of the project work concentrated on the characterization of OFHC copper brazed joints with copper-silver eutectic as filler metal. The following conclusions could be made from the experiments:

- i) The tensile strength of the joint is more than the strength of the base metal, and the failure occurs from the base metal.
- ii) The shear strength of the joint is also more than that of the base metal, given the fact that the overlap distance in a joint is more than twice the thickness of the thinnest portion of the workpiece. As the overlap distance becomes less than above, mentioned tearing at the joints is observed before failure and is indicative of the fact that the shear strength of the joint becomes less than that of the base metal.
- iii) Leak rate tightness of a brazed joint is more than 10^{-10} atm-cm³/sec.
- iv) The proper brazing which means proper flow of the filler metal through the joint surface depends on many factors such as clearance between the joints, quality of the furnace atmosphere, the load applied on the workpiece, the form and amount of filler metal, the soaking time and temperature, the brazing temperature, the design of the joint and most importantly the cleaning procedure.

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